Final Report on the World Trade Center (WTC) Dust Screening Method Study

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ACRONYMS:

ATSDR	Agency for Toxic Substances and Disease Registry
AVG	Average
COPC	Contaminant of Potential Concern
EPA	U.S. Environmental Protection Agency
EPIC	Environmental Photographic Interpretation Center
ERT	U.S. EPA's Emergency Response Team
HEPA	High Efficiency Particulate Air
LI	Long Island
MMVF	Man Made Vitreous Fibers
MQO	Measurement Quality Objective
ND	Non-Detect
NEIC	U.S. EPA's National Enforcement Investigations Center
NERL	U.S. EPA's National Exposure Research Laboratory
NJ	New Jersey
NYCDOMH	New York City Department of Health and Mental Hygiene
ORD	U.S. EPA's Office of Research and Development
PM	Particulate Matter
$PM_{2.5}$	Particulate Matter Smaller than 2.5 microns
QAPP	Quality Assurance Project Plan
SD	Standard Deviation
SEM	Scanning Electron Microscopy
USGS	U.S. Geological Survey
WTC	World Trade Center

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EXECUTIVE SUMMARY

The September 11, 2001 attack on the World Trade Center (WTC) covered a large area with dust and debris. To assist in determining if residual contamination exists in the indoor environment, the U.S. Environmental Protection Agency (EPA) initiated a study to sample indoor environments that may have been impacted by the WTC collapse. A critical component of this study is determining whether sampled dust originated from the collapse of the WTC or instead is urban dust originating from other sources. This report describes work performed to develop and validate a screening method for indoor dust that can be used to determine whether dust sampled is from the collapse of the World Trade Center towers.

Dispersion models, monitoring, photos, interviews, and satellite data were reviewed to discern areas that were likely impacted by WTC emissions and those that were not (US EPA 2002; 2004). A total of 117 samples were collected from both impacted and non-impacted areas. A subset of these samples were analyzed by EPA's National Exposure Research Laboratory (NERL) and National Enforcement Investigations Center (NEIC), and United States Geological Survey (USGS) to evaluate the slag wool levels in the dust and develop an analytical method. The analytical method that was developed screens for three materials that are believed to be present in large quantities in WTC dusts: slag wool, elements of concrete, and gypsum. This method involves the use of Scanning Electron Microscopy (SEM) to determine the quantity of each of the materials present.

Five commercial laboratories, along with the three above listed government labs, were recruited to test the screening method. Thirty-two dust samples, consisting of both confirmed background samples and a confirmed background dust spiked with varying amounts of confirmed WTC dust, were sent out to the eight labs. The labs were provided the samples "blind". They did not know which samples were background dust and which were non-impacted dust spiked with WTC dust. In addition to the thirty-two samples, one of the five commercial laboratories also received twenty-eight background samples to increase the available data characterizing background locations.

The data reported by these laboratories indicated the following:

1) Five of the eight laboratories were able to reasonably measure the slag wool concentrations in non-impacted dust spiked with confirmed WTC dust.

2) A substantial amount of variability in slag wool measurements was found within labs and between labs. Despite this variability, slag wool measurements appear to be sensitive enough to distinguish WTC dust (defined as 4 Albany) spiked at the 10% level from background dust.

3) The levels of gypsum and elements of concrete in the spiked samples were indistinguishable from the levels in the background samples. This suggests that, while these components may have been elevated in dust samples collected near the WTC site in September 2001 (as found by USGS in their studies on WTC dust), they are also commonly found in the indoor environment and would not be useful as WTC signature

components.

4) Analysis of samples during method development showed elevated levels of slag wool in samples from several impacted locations compared to slag wool levels measured at background locations.

I. INTRODUCTION AND BACKGROUND

The objective of this effort was to develop and validate a means of determining whether dust sampled as part of EPA's planned sampling program contains residual contamination attributable to the collapse of the WTC towers. The tested screening method is a critical component of the sampling program as it will be used for two primary purposes: 1) to determine the geographic extent of the dust remaining from the collapse impact, and 2) along with the results from contaminants of potential concern (COPC) testing, to determine the need for a clean-up of the sampled areas.

The USGS has published two reports that provided the basis for the initial hypothesis that a WTC collapse signature is comprised of three marker components: slag wool, gypsum and elements of concrete. The first report discusses the analysis and interpretation of indoor and outdoor WTC dust samples collected near Ground Zero, days and weeks after September 11, 2001 (Meeker et al., 2005). From this work, we see that the WTC dust samples are dominated by gypsum, concrete, and man-made vitreous fibers (MMVF), mainly slag wool. It is on the basis of these key results that gypsum, elements of concrete, and slag wool were identified as candidates for a WTC signature. The second report discusses the analysis of EPA supplied samples taken from several indoor locations well outside of the WTC impacted area (background). These samples were taken between September of 2004 and April of 2005. Slag wool was absent from many of these background samples, but Lowers et al. (2005a) state that the samples do have gypsum present, which they speculate might be due to the presence of wall board in the sampled apartments. Because of the lack of slag wool in these samples, USGS concluded that these samples did not contain WTC dust. USGS also concluded that perhaps slag wool is the single most critical of the three WTC dust constituents when distinguishing WTC dust from other common dusts.

Other studies also identified MMVF and gypsum as predominant components of WTC dust. In a study of air and settled dust quality in apartments in Lower Manhattan, the Agency for Toxic Substances and Disease Registry (ATSDR) and the New York City Department of Health and Mental Hygiene (NYCDOMH) found significantly more MMVF and gypsum in samples taken from Lower Manhattan apartments as compared to samples taken from apartments in areas above 59th Street (NYCDOMH/ATSDR, 2002). They also concluded that gypsum was seen at a higher percentage level in the Lower Manhattan dust samples as compared to the comparison area samples. In a comprehensive study of the composition of settled dust in the Deutsche Bank building at 130 Liberty Street, R.J. Lee identified numerous hazardous contaminants that were present in the dust at levels much higher than in background office buildings, and among those substances identified in their "WTC signature" were mineral wool and gypsum (R.J. Lee, 2004).

If the WTC building collapse signature components of slag wool, gypsum, and elements of concrete are not present, then one could conclude that WTC building collapse dust is not present. However, since these components might be present in typical New York City dust, and as slag wool is a component of insulating materials in currently constructed buildings, it is possible that a test might show them to be present even though WTC dust never impacted the sampled area. A 'screening test' will, by its design, result in some fraction of such false positives (i.e. a location without residual WTC dust that tests positive for the above components). However, an appropriate 'screening test' would result in very few, if any, false negatives (i.e. a location with residual WTC dust that tests negative for the above components).

II. METHOD DEVELOPMENT

Sample Collection

EPA acquired 117 dust samples during the time period of September 2004 to April 2005. Twenty-one 'impacted' samples were taken by the EPA at two buildings that were part of the Deutsche Bank complex located at 130 Liberty Street and 4 Albany Street. Both affected buildings were uninhabited and slated for demolition. Fifty samples were taken from locations well beyond the impacted zone (based on modeling, monitoring and photo analysis; these samples are considered to be 'background' dust). Forty-six samples were taken from locations that were possibly impacted, but were a bit farther from the WTC site than the known 'impacted' samples. None of these forty-six samples were used in the method validation study, but several were evaluated during both the method/protocol development phase and post-study. In addition, one impacted sample was obtained from the USGS. This sample was a composite sample of outdoor and indoor WTC dust collected in September of 2001.

A standard method utilizing a High Efficiency Particulate Air (HEPA) vacuum collector was used by EPA to collect most bulk dust samples. Information on this method is provided in the Quality Assurance Plan (QAPP) for this study (Appendix A). Some bulk dust samples were collected from residential and commercial vacuum cleaner bags.

Modeling and satellite photography were used to determine sampling locations for the collection of the 117 samples. Figures 1a and 1b (EPA 2002; EPIC 2004) are examples of modeling and photographic analysis used to distinguish non-impacted or background locations. Figure 1a shows ORD-modeled WTC Plume Dispersion on September 11, 2001 at 12 noon. The values indicated by red are hourly $PM_{2.5}$ concentrations (in $\mu g/m^3$) measured at pre-existing NJ and NY State-operated PM monitoring stations in northern New Jersey and New York City. Red, orange, and yellow shading represent most likely areas of plume dispersion (red = estimated dilution to 100th to 500th and dark blue = dilution to < one millionth of pollutant concentration at WTC source)**.** As seen in this figure, the plume very rapidly diluted to concentrations less than 1/1000 (which is the yellow area) of the initial source strength at Ground Zero. Figure 1b shows the boundaries of collapse deposition debris as determined by aerial photographs. This photograph was taken on September 13, and shows the four areas of "confirmed", "probable", "possible", and "no dust" from the collapse. These areas were used in the determination of strata used in the design for the overall sampling program.

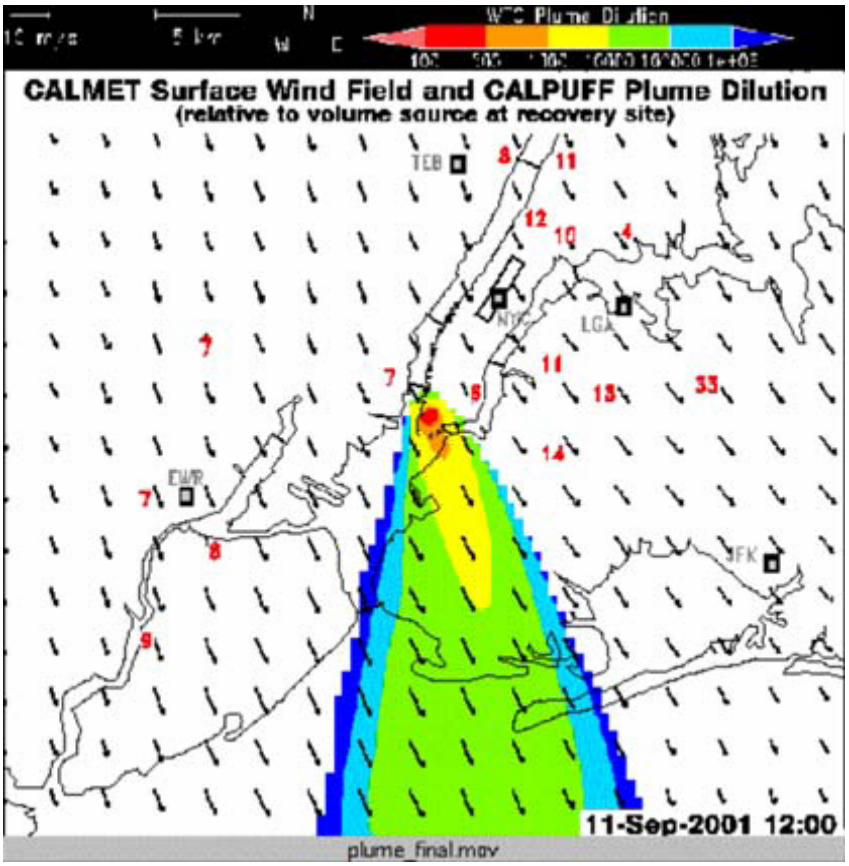

Figure 1a: ORD-modeled WTC Plume Dispersion on September 11, 2001 at 12 noon. (Source: Exposure and Human Health Evaluation of Airborne Pollution from the World Trade Center Disaster (External Review Draft). U.S. Environmental Protection Agency, Washington, D.C., 2002.)

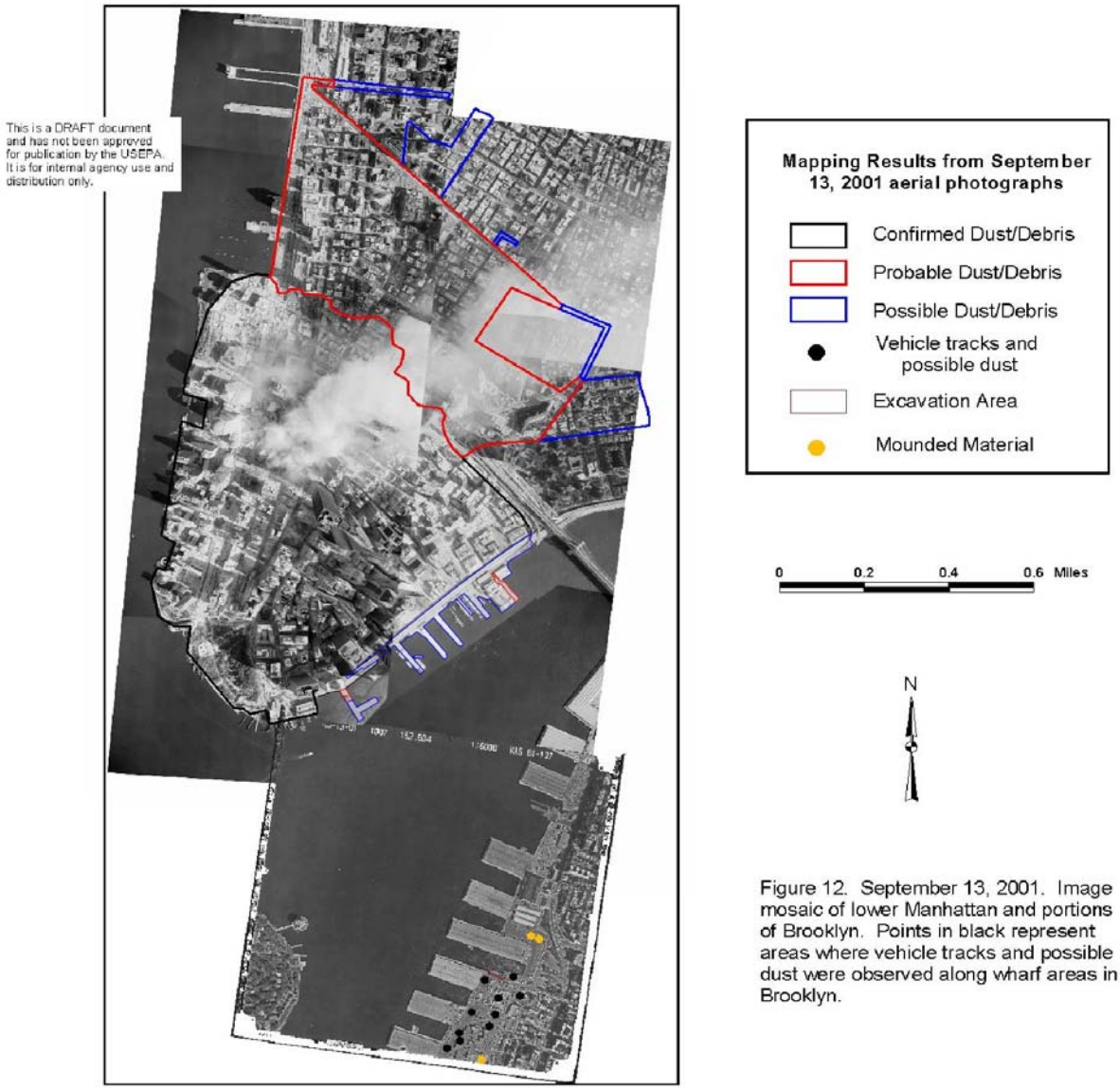

Figure 12. September 13, 2001. Image mosaic of lower Manhattan and portions of Brooklyn. Points in black represent areas where vehicle tracks and possible dust were observed along wharf areas in Brooklyn.

Figure 1b: Display of boundaries of expected deposition based on analysis conducted by EPA's Environmental Photographic Interpretation Center (Updated by EPIC from the figure which appears in EPIC, 2004).

Preliminary Analysis of Collected Samples for Slag Wool
Most of the collected samples were analyzed for slag wool content by the EPA's National Exposure Research Laboratory (NERL) Scanning Electron Microscopy (SEM) Laboratory. This analysis was performed as part of the EPA's development of a protocol for sample preparation and analysis and for preliminary sample characterization. These samples were not analyzed for elements of concrete or gypsum as an analytical method for these components had not yet been developed. The data acquired during this method/protocol development effort are presented in Appendix B. Caution should be used with these data as it was obtained while the method was being developed. Post-study data acquired by NERL are also presented in Appendix C.

In evaluating the method development data acquired by NERL (Appendix B), there appears to be a distinction between samples taken in impacted areas versus background samples. Eighteen of the 21 samples from impacted areas had slag wool at concentrations of greater than 100,000 slag wool fibers per gram of dust, with a range of 69,000 to 13,400,000, while all of the samples from background areas had concentrations less than 100,000 fibers/gram, ranging from no slag wool detected (in 12 of 47 samples) to 92,800 fibers/gram of dust.

Based on this preliminary work, the USGS, the EPA's Office of Research and Development (ORD), the EPA's National Enforcement Investigations Center (NEIC), and experts five commercial testing laboratories (denoted labs A-H in Appendix E), worked together to develop an analytical method to identify the presence and concentration of the screening constituents (i.e. slag wool, gypsum and elements of concrete) in indoor dust. This method was reviewed by the WTC Expert Technical Panel's signature subcommittee and is presented in Appendix D. The composition of this technical panel can be found at http://www.epa.gov/wtc/panel.

III. METHOD VALIDATION STUDY

Study Design
The basis for the WTC dust screening method discussed above is as follows: if a unit has been impacted, those materials that are found in WTC dust will be found in the dust collected from the unit. The materials under consideration are: 1) slag wool, 2) elements consistent with concrete and 3) gypsum. The study described herein was intended to validate the WTC dust screening method by demonstrating the following things:

1) that the above described materials are reasonable markers for WTC dust (by showing that these markers distinguish WTC-laden dust from background dust);
2) that WTC dust at a diluted concentration can be distinguished from background; and
3) that the analytical method works well enough and is able to be carried out by enough analytical laboratories to: 1) evaluate the above materials as markers and 2) distinguish WTC dust from background dust.

The first of these three objectives was partially addressed in method development work, which focused on slag wool. As described in the previous section, slag wool was found to be elevated in locations deemed "impacted", while slag wool was not detected or detected at low concentrations in "background" areas.

Five independent laboratories and three government laboratories participated in this method validation phase. One government laboratory analyzed only a small portion of the samples, but this lab was critical in the method development. Each laboratory attended a two day session during which the method was further developed and discussed, and the protocol was adapted to suit each laboratory's equipment.

Following this session, the laboratories received dust samples consisting of both confirmed background samples (10 samples plus duplicates for a total of 20) and confirmed non-impacted dust spiked with varying amounts of confirmed WTC dust (6 spiked samples plus duplicates for a total of 12). Specifically, a sample that was characterized and confirmed as non-impacted (designated in Appendix B as NE Queens maid service) was split, and the splits were spiked at levels of 1, 5, and 10% total mass with two different characterized and confirmed WTC dusts. These spiked samples were then homogenized as documented in the QAPP for this study (Appendix A). The two spiking dusts were 1) a composite sample of predominantly outdoor dust collected in September of 2001 by USGS, and 2) dust collected by the U.S. EPA from the Deutsche Bank building at 4 Albany Street in September of 2004. The 4 Albany Street building borders the south side of the WTC complex. Six spiked samples were prepared for each laboratory; these were split so that each laboratory received 12 spiked samples. Each laboratory also received 10 non-impacted background samples that were also split, resulting in a total of 20 background samples. Thirty-two samples in all were sent for analysis to the eight labs.

In addition to the 32 samples, one of the five commercial laboratories also received 28 background samples to increase the available data characterizing background locations.

The labs were provided the 32 samples "blind"; they did not know which samples were pure background dust, and which were the spiked dust. To ensure sufficient results for spiked samples, the government laboratory that was only able to analyze a small portion of the samples was asked to analyze only the 12 spiked samples. Again, they were not told the identity of these samples (Lab C). The labs had five weeks to analyze all samples. The final data from all laboratories, including the data for the additional 28 background samples, were reviewed, evaluated and analyzed by the EPA and the EPA's prime contractor. This prime contractor's from this analysis is presented in Appendix E.

Composition of Spiked Samples

The USGS performed an analysis of the spiked, homogenized samples prior to the samples being sent to the labs. The measured levels were in the approximate range for the spiking percent (1, 5, and 10%) based on the undiluted concentration level of each WTC dust and, in all but one case, each percent level was fully distinguishable from the others (Figures 2 and 3). The variability in the measured levels was expected due to the difficulty in homogenizing dusts that have large particle size distributions, and the fact that components of WTC dust will vary within a sample because of the nature of the source. Given these difficulties and the measurement results, these dusts were determined to be reasonably homogeneous.

As seen in Figures 2 and 3, the level of slag wool differs between the two WTC dusts, with the pure dust that was collected from 4 Albany Street in 2004 more than an order of magnitude lower than the dust collected by the USGS in September of 2001. The pure dust from 4 Albany

Street had slag wool levels at 500,000 fibers/gram of dust versus approximately 11,000,000 fibers/gram of dust for the USGS collected sample. There are likely explanations for this large difference in slag wool levels. The USGS sample was a composite of multiple outdoor samples and one indoor sample taken during September of 2001. The 4 Albany was an indoor sample was taken three years post 9/11 in September of 2004. As this 4 Albany sample was taken exclusively inside of a building, it was not only diluted by three years accumulation of urban background dust, but was also characteristic of dust that had penetrated the shell of a building as opposed to that deposited on the ground outside.

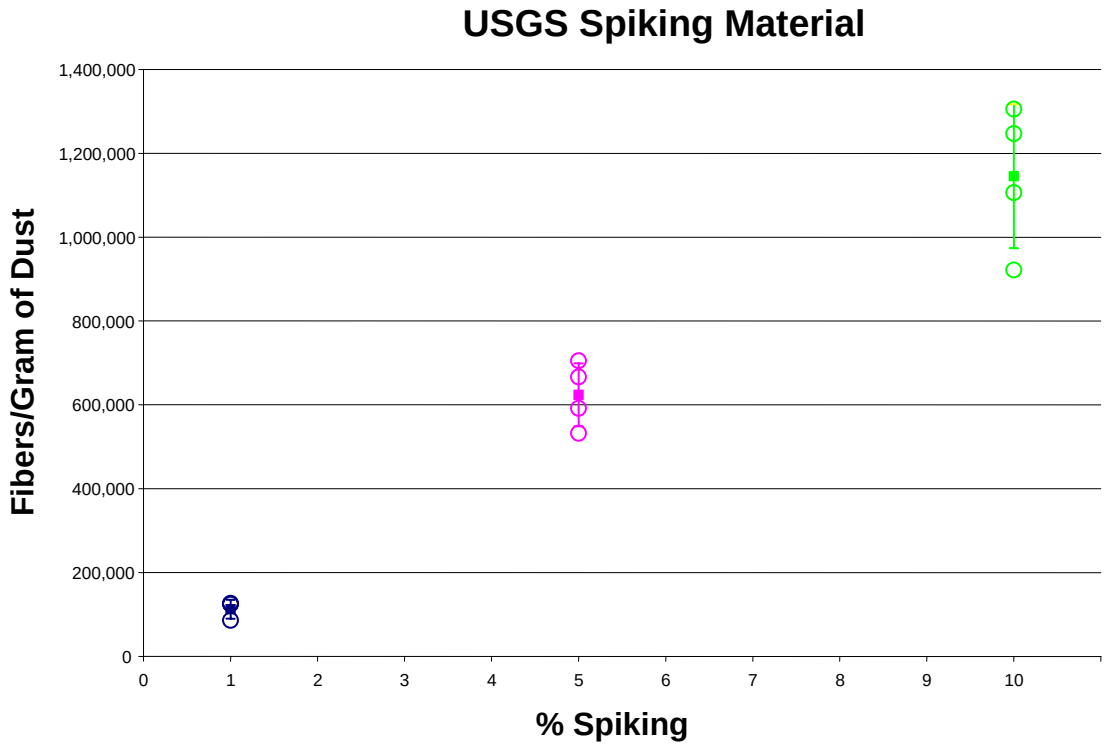

Figure 2: USGS Spiking Material Results. Analysis was conducted by USGS prior to being sent to labs for study. Pure dust averaged approx. 11,000,000 fibers/gram. (Figure provided by USGS)

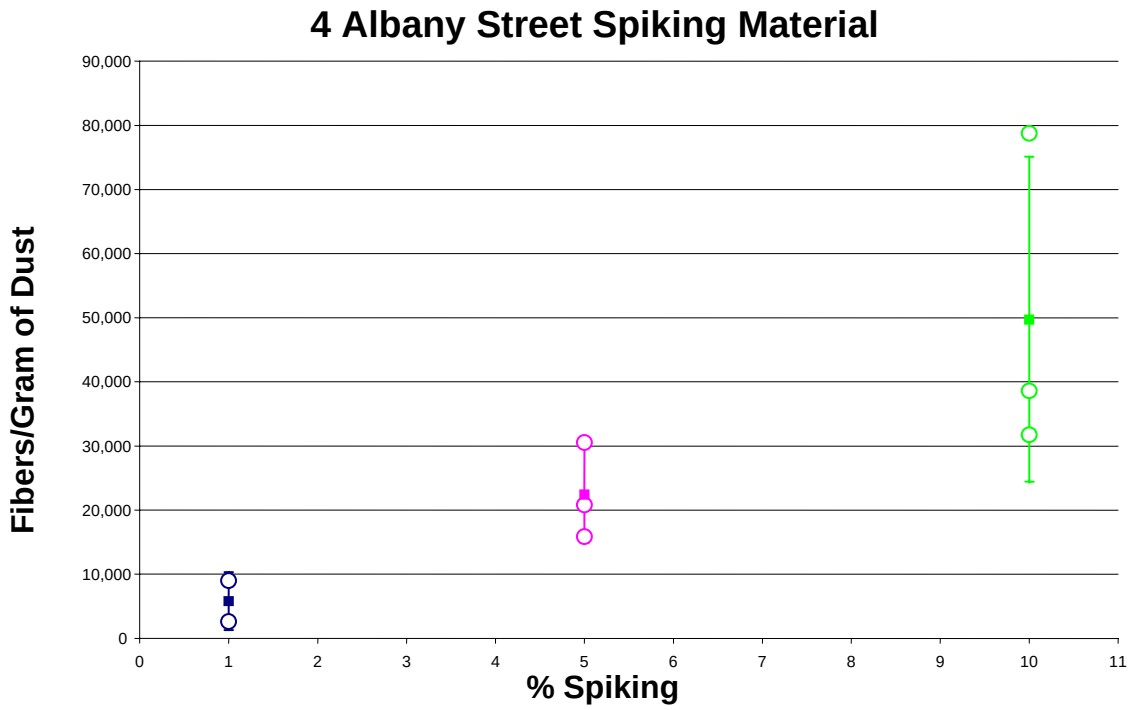

Figure 3: 4 Albany Street Spiking Material Results. Analysis was conducted by USGS prior to being sent to labs. Pure dust averaged approx. 500,000 fibers/gram. (Figure provided by USGS)

IV. RESULTS AND DISCUSSION

Development of Study Results

The final report from the prime contractor with all raw analytical and calibration data can be found in Appendix E. A summary of the study results that includes the data from the 28 additional background samples analyzed by a single commercial laboratory is provided in Table I, as well as Figures 4-7. A map of the origin of the samples analyzed during this study is shown in Figure 8.

All background sample data used in Table I and Figures 4-7 are from the Greater NY City area. Background samples taken in Research Triangle Park, North Carolina are not included as they are not representative of NY City background dust. Data for all background sample results may be found in Tables 3 and 4 of the Versar report in Appendix E. It should be noted that the Research Triangle Park samples show higher slag wool levels than NY City area background samples. This is due to the presence of slag wool containing ceiling tiles in the building sampled. Note also that Table I indicates two average values for background slag wool. These values reflect the inclusion and exclusion of two samples collected in New Jersey (NJ) and Long Island (LI) that were extremely high in slag wool fibers, likely due to their insulation, fireproofing or ceiling tiles. Based on these results it is likely that some false positive results will occur in buildings with slag wool-based ceiling tiles, fireproofing or insulation. .

Three of the commercial laboratories, designated as labs E, F and G, reported analytical data that are not consistent with other five labs. Generally, these labs were not able to distinguish differences between the three spiking levels. In addition, these labs did not meet the measurement quality objectives (MQOs) for the spiked samples put forth in the QAPP for this study (Appendix A Section A.7.1). Thus, the data from these three labs are not considered in the results presented in Table I and Figures 4-7. The statistical analysis performed to make this determination is presented in Appendix F. In addition, Lab H was not considered when determining concrete and gypsum levels as their data were at least two times higher than the sample average without these data (Table I and Figures 6 and 7).

In discussions with the commercial laboratories, it was determined that some labs did not have the personnel or the equipment to perform the required analysis in the given timeframe, thus, data quality became an issue. Additionally, labs that had less experience with slag wool analysis felt that a clearer definition, in addition to that provided in the catalog developed by USGS in Lowers et al., 2005b, of slag wool was needed to distinguish it from other mineral wools. Finally, labs that were unable to automate the gypsum and concrete analysis expressed their belief that the method was too long and complicated for accurate quantitative dust analysis. All laboratory comments will be taken into consideration in when finalizing the protocol.

	Background (Greater NY Area)	USGS Spiked (Collected 9/01)	4 Albany Spiked (Collected 9/04)
Slag Wool Average (fibers/g dust)	*AVG ± SD* 35,950 ± 74,300 17,740 ± 15,835* *Range of Samples* ND* - 369,230 ND* - 60,000**	*1%* 94,000 ± 25,740 *5%* 452,510 ± 100,640 *10%* 870,280 ± 310,420	*1%* 17,270 ± 7,880 *5%* 52,510 ± 26,140 *10%* 88,540 ± 18,300
Elements of Concrete (% Area)	*AVG ± SD* 15.6 ± 5.7 *Range of Samples* 6 – 30.5	*1%* 20 ± 6 *5%* 19 ± 7 *10%* 16 ± 2	*1%* 15 ± 1 *5%* 18 ± 4 *10%* 16 ± 3
Gypsum (% Area)	*AVG ± SD* 9.5 ± 3.4 *Range of Samples* 4 – 16.5	*1%* 9 ± 6 *5%* 7 ± 3 *10%* 6 ± 0.5	*1%* 9 ± 4 *5%* 5 ± 2 *10%* 7 ± 2

- **ND=Non Detect (Zero slag wool fibers)
- *Two extremely high values from NJ and LI removed.

Table 1: Avg, Standard Dev., and Range of Results for Background and Spiked Samples (Data Summarized from Tables 1, 2, 3 and 4 of Appendix E).

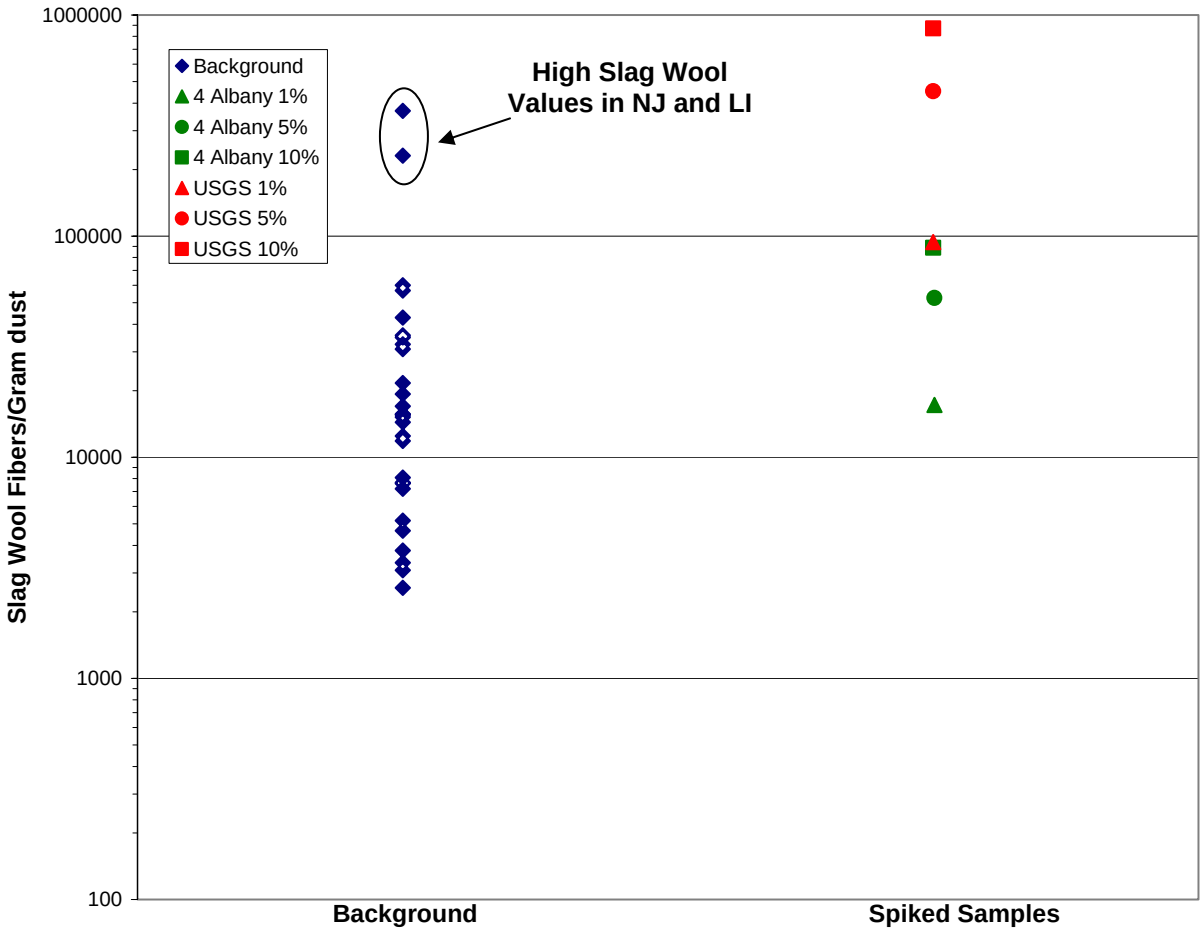

Figure 4: Average Slag Wool (Fibers/Gram of Dust) in background and spiked samples. (Data from Tables 3 and 4 Appendix E)

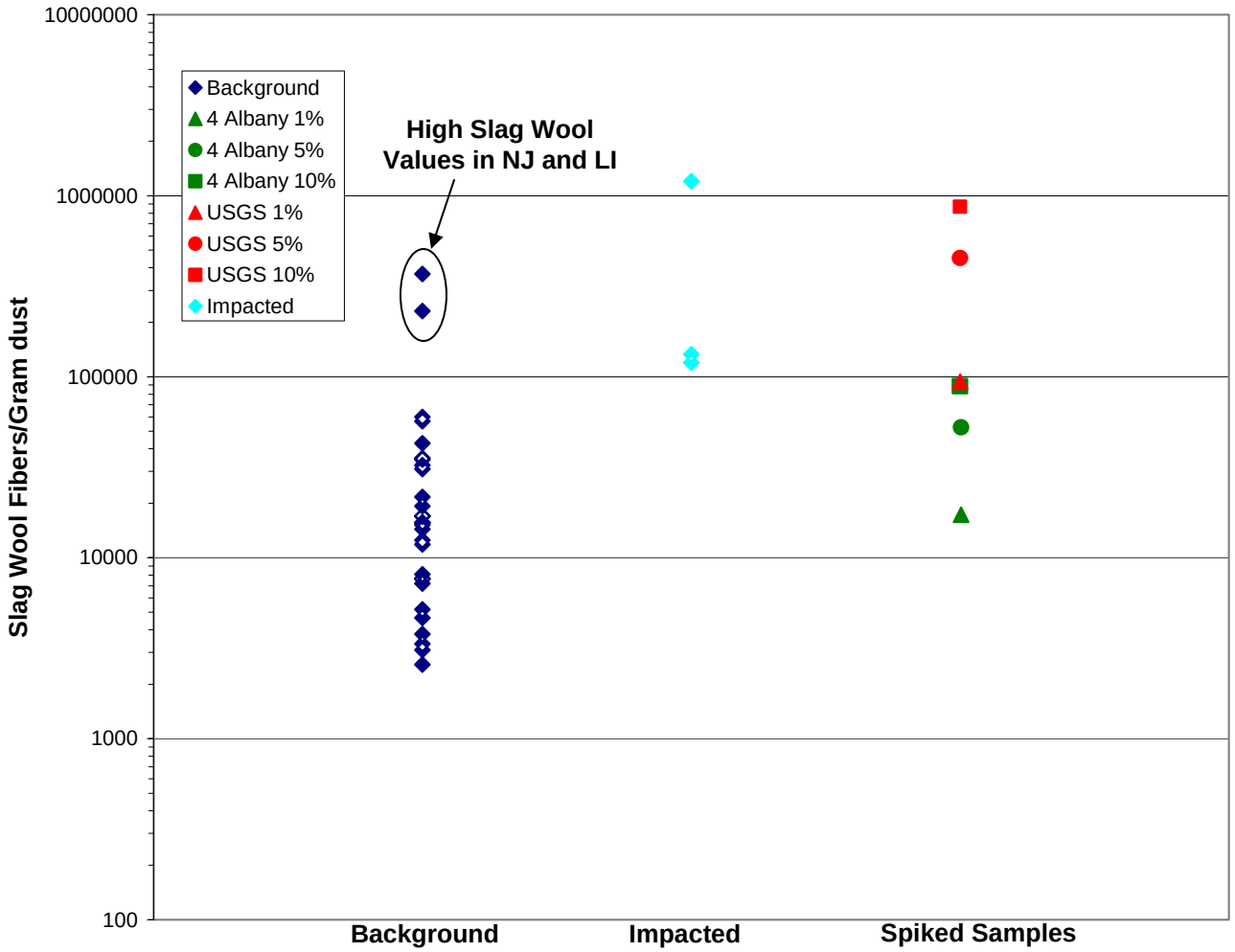

Figure 5: Average Slag Wool (Fibers/Gram of Dust) in background, spiked and impacted samples. Impacted samples are locations that are shown in satellite pictures to have been affected by WTC Collapse Dust. Slag wool results for impacted samples were derived during method development and were not part of this method validation; they are provided for comparative purposes. These impacted samples range from 0.1 to 1.6 miles from the WTC site (see Figure 8 for sample origin location). Data from Appendix B (Impacted) and Tables 3 and 4 of Appendix E (Background and Spiked).

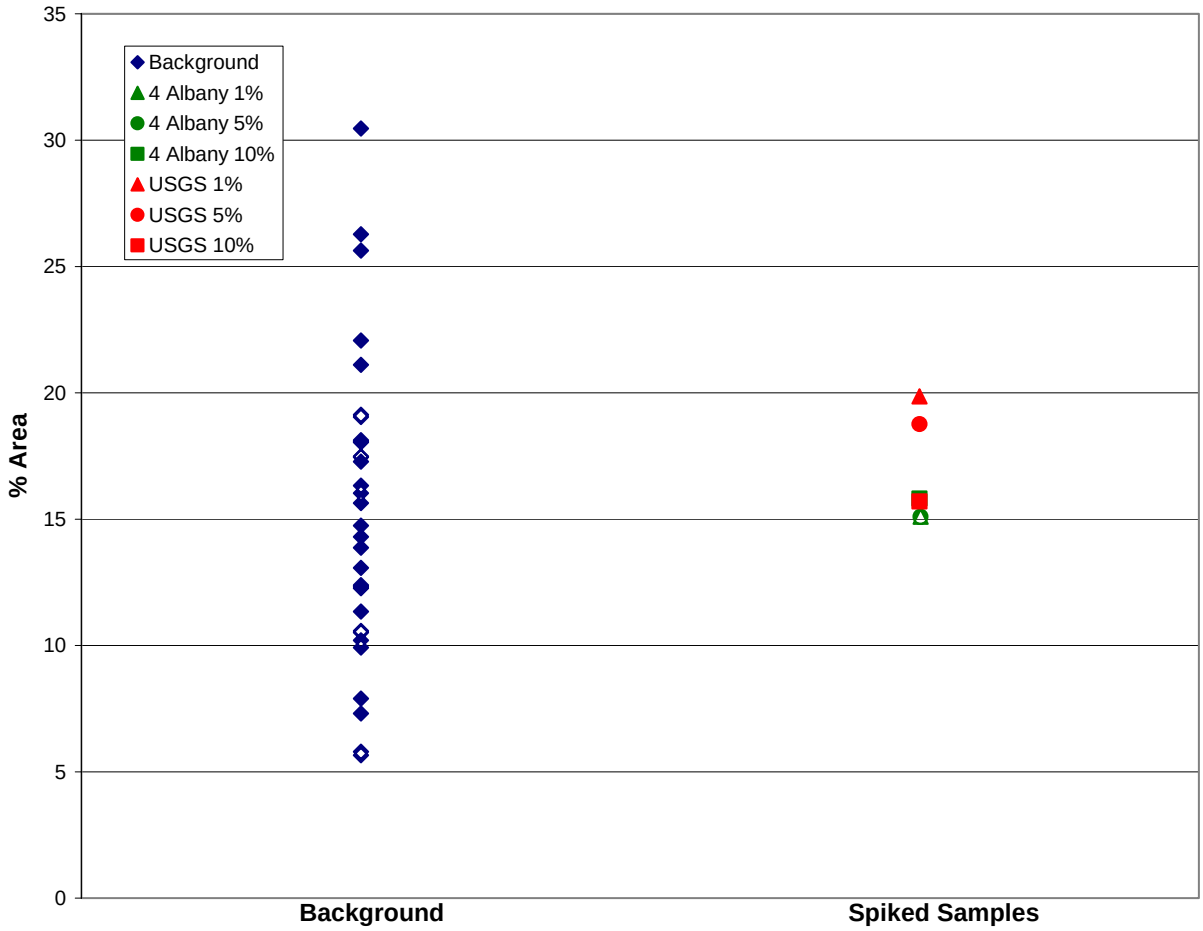

Figure 6: Average of Elements of Concrete (% Area) in background and spiked samples. (Data from Tables 1 of Appendix E)

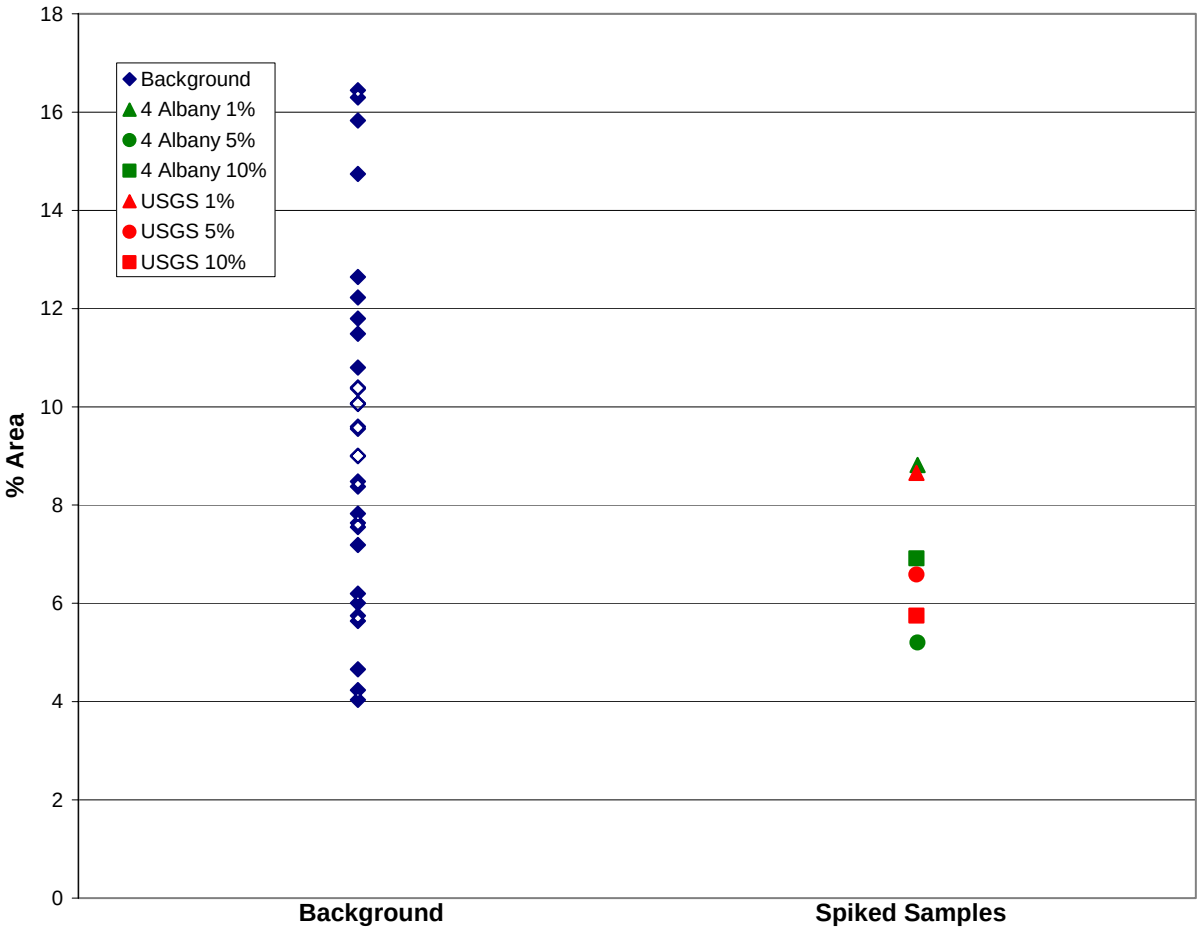

Figure 7: Average of Gypsum (% Area) in background and spiked samples. (Data from Tables 2 of Appendix E)

DESIGNATION	LOCATION
1,2	Stony Brook, LI
3,4	West End Ave between 72nd and 73rd Streets
5,6	30th Ave between 21st and 23rd, Queens
7	70th Street between 20th and 21st Ave, Brooklyn
8	79th St between York and East End Ave, Manhattan
9	92nd Street between Columbus and CPW, Manhattan
10	Columbia Medical Center, W. 168th St, Manhattan
11,HS3(1),HS(2)	Teaneck, NJ
14,15	88th Street between Amsterdam and Columbia, Manhat
16	80th Street between Riverside and East End Ave, Ma
17,MW(1),MW(2)	West End Ave between 105th and 106th Streets
18,19,20,21,22,23,C1-RTP1+2	Research Triangle Park, NC
24,25,26	Edison, NJ
AP5(1),AP5(2)	Chittenden Avenue, Manhattan
CMC(1),CMC(2)	Columbia Medical Center, W. 168th St, Manhattan
WGS(1),WGS(2)	Nassau County, LI
USC(1),USC(2)	Federal Courthouse, White Plains, NY
MUNYC1(1)+(2),MUNYC2(1)+(2)	Northern Manhattan, Above 70th Street
12,13	Nassau County
28	NE Queens
27	Long Beach Island, NJ
FP(1),FP(2)	Federal Courthouse, Central Islip, NY

Figure 8: **Map of the origin of the samples analyzed during this study (Reference Appendix D for sampling data).**

Discussion

Slag wool appears to be an indicator for WTC dust and can be distinguished from background dust at all three spiking levels for the USGS dust and at the 10% level of the 4 Albany Street dust. The 4 Albany Street dust is considered to be WTC impacted dust but as noted earlier, the 4 Albany dust likely had lower levels of slag wool due to the fact that it was an indoor dust that was not sampled until three years after the WTC collapse.

Levels of gypsum and elements of concrete have no discernable relationship to the level of WTC dust. There does not appear to be a distinguishable difference between levels of concrete and gypsum in background dust and the samples spiked with WTC dust, despite USGS analysis of WTC dust from 2001 (Meeker, 2005) showing elevated levels of these components. This is likely due to the fact that while these components may seem high in WTC dust, they are also high in general background dust as they are common building materials.

While method development (Appendix B and summarized in Section II above) work showed that dusts from known impacted locations generally had slag wool levels above 100,000 fibers/gram, several samples taken within this impacted zone and analyzed during method development showed lower levels of slag wool. Two likely explanations can be offered for these results. First, as the data in Appendix B was acquired during method development, it must be viewed as such, and second, multiple cleanings of the inhabited areas since September 11, 2001 may have removed residual WTC collapse contamination. The majority of these samples were taken in fully inhabited buildings, from locations within the buildings that can be characterized as either 'accessible' or 'infrequently accessed' areas. These terms are described in the final draft EPA sampling program, and they denote areas that are accessed by people over the course of time, such as counter tops or rugs (accessible) or underneath furniture (infrequently accessed). For this reason alone, it is encouraging that a substantial amount of the dust sampled in late 2004 and beyond had high levels of slag wool.

While there was ample evidence of higher levels of slag wool associated with the WTC dust and lower levels associated with background, there is high variability in slag wool measurements within and between labs. Estimates of within lab relative standard deviations based on analysis of duplicate samples of the 4 Albany Street data are 55%, 24% and 14% for the 1%, 5% and 10% dilution levels, respectively. Estimates of between lab relative standard deviations based on the 4 Albany Street data are 64%, 70% and 29% for the 1%, 5% and 10% dilution levels, respectively (looking at results from analysis of the same spike level samples by multiple labs). Causes of the high levels of variability may include:

- Procedures to homogenize the spiked samples did not result in complete mixing and distribution of fibers; they instead resulted in a 'reasonably' homogeneous sample given the large size variation of the dust components.
- Components of both non-impacted/background and WTC dusts will vary within a sample because of the inherent nature of the dust samples. Thus, the samples received by the labs may vary in content.
- Operator experience with the target components appeared to be an issue – post-study discussion indicated that labs representatives with less familiarity with slag wool expressed a belief that further guidance as to its definition was needed.

- The variability in the mass of dust used for the analysis, as the protocol allows for a range, not a specific mass, to be used. This range is essential due to the extreme differences in slag wool levels possible between background and spiked samples.

Finally, it is noted that Table I indicates two average values for background slag wool. These values reflect the inclusion and exclusion of two samples (and their duplicates) collected in New Jersey (NJ) and Long Island (LI) that were extremely high in slag wool fibers, likely due to their insulation, fireproofing or ceiling tiles. Similarly, it was earlier noted that samples taken from a North Carolina building due also to slag wool used in ceiling tiles were not included in the interpretative analyses. Based on these results, it is likely that some false positive results will occur in buildings with slag wool-based ceiling tiles, fireproofing or insulation.

V. CONCLUSIONS

The interlaboratory results indicate that the better performing labs are capable of distinguishing the difference between 1, 5 and 10% 4 Albany Street dust. Also, despite the high levels of within sample and within lab variability, the method using slag wool appears to be sensitive enough to distinguish 10% 4 Albany Street dust from background dust. Additional evaluation of the data will be performed to further understand the variability. Measures will be taken (i.e. standards will be sent regularly to each lab) during EPA's planned sampling program to evaluate the accuracy and precision of the laboratories.

In summary, the data developed in this study support the following findings:

1) Five of the eight laboratories were able to reasonably measure the slag wool concentrations in background dust spiked with confirmed WTC dust.

2) High levels of variability in slag wool measurements, both within labs and between labs, were observed in the data. Despite this variability, the slag wool method appears to be sensitive enough to distinguish WTC dust from background dust at the 10% level (defined as, 4 Albany Street).

3) The levels of gypsum and elements of concrete in the spiked samples were indistinguishable from the levels in the background samples. This observation suggests that, while these components may have been elevated in dust samples collected near September 2001, as found by USGS in their studies on WTC dust, they are also commonly found in the indoor environment and would not be useful as WTC signature components.

4) Analysis of samples during method development generally showed slag wool levels in samples from impacted locations to be greater than slag wool levels in samples from background locations.

VI. REFERENCES:

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IX. APPENDICES

APPENDIX A: QUALITY ASSURANCE PROJECT PLAN FOR THE WORLD TRADE CENTER (WTC) SCREENING METHOD STUDY

(Due to formatting - this document will be provided under separate cover)

APPENDIX B: DATA ACQUIRED BY EPA NERL DURING METHOD DEVELOPMENT

Samples Collected at Background Locations

	slag wool fibers/ gram of dust	Average of Duplicates (slag wool fibers/gram dust)
Residential		
West End Ave between 72nd and 73rd Streets, Manhattan	2.53E+04	
	5.47E+04	
30th Avenue between 21st and 23rd St, Queens	2.80E+04	
	2.26E+04	
E 79th Street between York and East End Ave, Manhattan	4.93E+04	
Chittenden Avenue, Manhattan	1.53E+04	
	2.87E+04	2.20E+04
92nd Street between Columbus and CPW, Manhattan	2.42E+03	
80th Street between Riverside and West End Ave, Manhattan	1.46E+04	
Edison, NJ	0.00E+00	
Stony Brook, LI	1.79E+04	
	2.90E+04	
70th Street between 20th and 21st Ave, Brooklyn	4.09E+04	
	4.77E+04	4.43E+04
Teaneck NJ	0.00E+00	
Long Beach Island, NJ	0.00E+00	
West End Avenue between 105th and 106th Streets, Manhattan	1.77E+04	
Edison, NJ	4.12E+03	
88th Street between Amsterdam and Columbia, Manhattan	8.35E+03	
	0.00E+00	
	5.74E+03	
North East Queens (Maid Service)	0.00E+00	
	0.00E+00	
	0.00E+00	
	5.37E+03	
	1.02E+04	
	1.27E+04	
	0.00E+00	
	1.63E+04	
	6.43E+03	
	0.00E+00	
	1.65E+04	
	0.00E+00	
Nassau County, Long Island (Maid Service)	0.00E+00	
	1.95E+04	
Business		
Port Authority Bldg, Port of Newark, NJ	3.86E+04	
	3.45E+04	
	7.32E+04	
	5.09E+04	
	1.85E+04	
	6.60E+04	

	8.58E+04	
Columbia Medical Center, W 168th St., Manhattan	0.00E+00	
	1.33E+04	
Edison, NJ	9.09E+04	
Federal Courthouse, Quarropas St, White Plains	9.28E+04	
Federal Courthouse, Islip, Long Island	9.00E+04	

Samples Collected at Known Impacted Locations

Business

290 Broadway, Manhattan	6.92E+04	
	8.81E+04	
	1.64E+05	
	1.95E+05	1.20E+05
	8.35E+04	
Broadway between Maiden Lane and John Street, Manhattan	1.33E+05	
Deutsche Bank Bldg, 130 Liberty Street, Manhattan	2.79E+05	
	4.71E+06	
	5.77E+06	
	6.60E+06	6.19E+06
	1.18E+07	
	1.22E+07	
	1.13E+05	
	2.06E+05	
	2.14E+05	
	2.25E+05	2.30E+05
	2.28E+05	
	2.78E+05	
Deutsche Bank Bldg, 4 Albany Street, Manhattan	6.36E+05	
	1.67E+06	
USGS Composite Sample Collected Sept 2001	1.34E+07	

Samples Collected at Locations with Unknown Impact

Residential

John Street between Gold and Pearl, Manhattan	1.26E+04
South End Avenue between Albany and Liberty, Manhattan	9.17E+03
River Terrace, Manhattan	0.00E+00
40th Street between Tunnel Exit St and 2nd Ave, Manhattan	2.91E+03
Orange Street between Henry and Hicks, Brooklyn	1.11E+04
24th Street between 8th and 9th Ave, Manhattan	3.32E+03
Montague between Montague Terrace and Hicks Street, Manhattan	5.03E+03
Houston and Mulberry Streets, Manhattan	6.30E+03

Business

Port Authority Bldg, Columbia St, Brooklyn	2.06E+05
	9.89E+04
	1.30E+05
	1.94E+05
	1.12E+04
	3.06E+05

Governor's Island	5.07E+04
	5.75E+05
	8.79E+04
Varick Street, Manhattan	9.57E+04

Samples Collected Outside of NY City

Business

Research Triangle Park, NC	5.00E+04
	8.96E+04

APPENDIX C: DATA ACQUIRED BY EPA NERL POST-STUDY

Samples Collected at Background Locations		
Residential		
Composite –North East Queens (Maid Service)	1.06E+04	
	1.49E+04	1.28E+04
Business		
Port Authority – Port of Newark, NJ	9.77E+03	
Samples Collected at Impacted Locations		
Business		
Governor's Island	1.93E+04	
	6.39E+05	
	1.21E+06	
Port Authority Bldg, Columbia St, Brooklyn	1.22E+05	

APPENDIX D: PROTOCOL USED FOR THE SCREENING METHOD STUDY

Protocol for Preparation and Analysis of Residential and Office Space Dust by Polarized Light Microscopy and Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy

June 27, 2005

Prepared by:
U.S. Environmental Protection Agency
National Enforcement Investigations Center/ National Exposure Research Laboratory/National Homeland Security Research Center
Denver, CO and Research Triangle Park, NC

The use of trade names does not imply endorsement and are used for illustrative purposes only.

Contents

1.0 Purpose

This document describes sample preparation and analytical screening procedures for bulk samples of dust collected from residential and commercial office environments. These methods are collectively referred to as the protocol.

2.0 Scope/Application

The protocol describes polarized light microscopy (PLM) and scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS) to screen bulk dust samples for mineral slag wool, particles consistent with concrete compositions, and gypsum. The analysis methods include operating parameters and particle identification criteria.

2.1 Limitations of the Method and Future Considerations

This protocol provides a means of analyzing for particles consistent with those found in dust present after the collapse of the World Trade Center (WTC) in New York City. Components of WTC Dust have been documented and catalogued by the U.S. Geological Survey Denver Microbeam Facility and the images and characteristics shall be used in identification of particles (1).

The x-ray mapping procedure in sections 12.2.3 and 12.2.4 and the calculations presented in section 13.0 only determine the maximum percentage of non-gypsum, calcium-rich particles, which may include non-concrete materials. The particle analysis procedure presented in section 12.2.5 is the preferred procedure for determining the percentages of gypsum and concrete particles in the sample.

The x-ray mapping and image analysis procedure relies heavily on the thresholds for backscattered electron images. Binary (particles white and background black) backscattered electron images (BEI) should be used to reduce errors in setting thresholds in Photoshop

3.0 Definitions

1. PLM – Polarized Light Microscopy
2. SEM – Scanning Electron Microscope
3. EDS – Energy Dispersive Spectrometry
4. SEI – Secondary Electron Image
5. BEI – Backscattered Electron Image
6. Mineral Wool – lightweight vitreous fibrous material composed of rock wool and slag wool and used especially for heat and sound insulation
7. Rock Wool – a man-made vitreous fiber (MMVF) component of mineral wool containing magnesium, aluminum, silicon, and calcium. Sodium and potassium may also be present. Iron oxide is typically 3-12% by weight.
8. Slag Wool – a man-made vitreous fiber (MMVF) component of mineral wool containing magnesium, aluminum, silicon, and calcium. Sodium and potassium may also be present. Iron oxide is typically less than 2% by weight.
9. HEPA – High-Efficiency-Particulate-Air Filter

4.0 Summary of Method

1. Weigh sample to nearest 0.0005 g.

2. Split the sample, archive half and keep half for analysis.
3. Ash half of the sample for analysis.
4. Sieve the ashed sample to 150 µm.
5. Split the <150 um ashed portion. Archive three quarters of the sample. Keep one quarter for PLM and SEM/EDS analysis.
6. Weigh the quarter and place it in enough isopropanol to get a 10-20 mg per mL dilution. Apply an aliquot to a glass slide, let dry, and add 1.55 (or 1.605) refractive index oil. Analyze by PLM for mineral wool.
7. Prepare a sample for SEM/EDS analysis using the same dilution prepared for PLM.
8. Apply an aliquot of the sample to an aluminum sample stub with a carbon adhesive tab covered by a piece of polycarbonate filter (13-mm diameter or punched out of a larger filter to fit the size of the stub).
9. Identify fibers by EDS and record the occurrence of fibers > 25 µm in length at 100 x magnification to get a statistical representation of fiber compositions.
10. Prepare 10-fold dilution of the suspension from step 7 and apply an aliquot to a polycarbonate/adhesive tab substrate affixed to an aluminum sample stub. Alternatively, a lighter loading can be prepared by filtering the diluted suspension through a 25-mm diameter, 0.4-µm pore size, polycarbonate filter and affix this to a carbon adhesive tab affixed to an aluminum sample stub.
11. Collect x-ray maps of 10 fields at 500 x magnification for major elements, especially Ca, S, and Fe and use Adobe Photoshop or similar software to determine the area percent of gypsum and Ca-rich particles. Fe-rich particles may also be identified in this step.
12. Perform particle analysis via computer-controlled SEM/EDX analysis.

5.0 Interferences

Interferences include possible contamination of samples by airborne dust or through improperly cleaned glassware and sieves. Interferences are minimized by performing all procedures involving dry dust in a clean room, cleaning countertops and glassware thoroughly before proceeding and placing particle-free wipes on all working surfaces. To avoid cross-contamination, properly clean all glassware, sieves, and tools between samples.

6.0 Safety

Respirable particles which may present a health hazard may exist in the sample. Bulk samples may release respirable particles during handling. All procedures involving dry dust samples will be performed under a negative flow High-Efficiency-Particulate-Air Filter (HEPA) hood. Samples handled outside of the HEPA hood will be covered with aluminum foil or placed in sealed glass jars.

7.0 Apparatus and Materials

1. HEPA negative flow hood
2. Forceps
3. Kimwipes
4. Stainless steel spatula
5. Weighing paper
6. Programmable furnace [not required for validation study]
7. Ceramic crucibles with lids [not required for validation study]

8. Analytical balance (accuracy to 0.0005 g)
9. Retsch ultrasonic sieve shaker (AS200 Basic), or similar [not required for validation study]
10. Sample sieves, 3-inch diameter (recommended), 150-µm (100-mesh) opening, with lid and bottom pan similar [not required for validation study]
11. SEM aluminum sample stubs
12. Conductive carbon adhesive tabs
13. Eppendorf pipette, 10-µL capacity
14. Disposable pipette tips
15. 1 – 10 mL pipette
16. Glass vials for sonicating dust in isopropanol suspension (holds 10-mL volume)
17. Razor blade
18. Ultrasonic bath
19. 50 mL glass beaker
20. Polycarbonate filters (25-mm diameter, 0.4-µm pore size)
21. Polycarbonate filters (13-mm diameter, 0.4-µm pore size), or borer to cut larger filters to SEM stub size
22. 11-mm diameter cork borer
23. Millipore filter apparatus for use with 25 mm filters
24. 125 mL Nalgene bottles
25. Hand-held vacuum pump
26. High-vacuum carbon evaporator with rotating stage
27. Glass etri dishes with lids
28. Adobe Photoshop Software, or similar
29. Glass petrographic slides
30. Glass cover slips
31. Polarized light microscope for mineral identifications
32. Scanning Electron Microscope with the following attributes:
    a. Resolution: 5 nm (at 25 kV, WD=10 mm – system dependent) or better
    b. Accelerating Voltage: 10 to 20 kV
    c. Minimum magnification range: 50x to 200,000x
    d. SEI (secondary electron image)
    e. BEI (backscattered electron image)
    f. Energy dispersive x-ray detector and analyzer for EDS analysis
    g. Ability to collect x-ray maps or particle analysis software (preferably both)

8.0 Reagents

1. Isopropanol, reagent grade [CAS No. 67-63-0]
2. 1.55 or 1.605 Refractive Index Oil

9.0 Sample Storage

Dust samples will be stored in an air-tight container, such as a sealed glass jar. Samples placed in reagents will be labeled appropriately and stored according to laboratory safety standards. Samples prepared for analyses will be stored in a protective container, such as a plastic case or covered etri dish, to prevent contamination.

10.0 Quality Control

Quality control is implemented by thoroughly cleaning glassware and spatulas, keeping working surfaces clean, and preventing cross contamination. During ashing, particles may be suspended if slow heating is not achieved. Following the ashing program as outlined will minimize flashing, which can cause particles to become airborne. Covered crucibles will be used to prevent contamination caused by flashing. Used Eppendorf pipette tips and weighing papers will be discarded and new tips and papers will be used for each

sample.

Duplicate samples shall be prepared to determine the precision of the analysis. In addition, sample blanks shall be prepared. These blanks are checks for cross contamination during handling of the samples. Blanks shall be prepared at the same time and in the same manner as samples.

10.1 Calibration

Calibration of the EDS system must be completed at least once at the beginning and again at the end of each analytical session. Backscattered electron image (BEI) calibration should be performed at the beginning of the session and anytime the backscattered image brightness and/or contrast is adjusted.

EDS calibration for both qualitative and quantitative (not required by this method but could be useful for identification of particle type) analysis is accomplished by the analysis of a polished carbon-coated reference standard. The recommended material is USGS BIR1-G basalt glass mounted in epoxy in a brass tube, polished, and carbon coated using a carbon evaporator (2, 3).

The calibration reference material should be analyzed at the same operating conditions to be used for the analysis including beam current, accelerating voltage, working distance, detector dead time, and sample tilt (= 0°). For BIR1-G the analysis should be performed with a beam size of 10-20 μm or equivalent area raster. All calibration spectra will be saved with the corresponding data set. The calibration data will be used for inter- as well as intra-laboratory comparisons. This calibration is in addition to, and not a substitute for the normal EDS calibration recommended by the EDS manufacturer which will be performed at regular intervals as specified by the EDS manufacturer.

Backscattered electron detector calibration can be performed on the same BIR1-G material by adjusting the detector brightness and contrast to achieve the following conditions. The epoxy on the BIR1-G reference material will be at 0 in a 256 grayscale image and the brass mounting tube will be at 256. The BIR1-G basalt glass should fall at approximately 130-140 gray scale units

11.0 Procedure

11.1 Weighing and Splitting

Weighing and splitting should be performed under a negative flow HEPA hood. If the fan speed is set too high, loss of particles may occur. The fan speed may need to be adjusted to prevent the loss of fine particles.

Obtain an analytical balance with an accuracy of 0.0005 g and preweigh a clean piece of weighing paper. Transfer the dust from the sample vial to the weighing paper and determine the weight of the dust. Split the sample with a clean razor blade using the cone-and-quarter method. If there are large clumps of organic fibers, such as hair or lint, temporarily remove the hair with a pair of forceps and tap the forceps lightly with another tool over a piece of weighing paper to remove fine particles. Center the fine fraction on the paper and split the sample into four equal parts using a razor blade. Collect opposite corners (½ of the sample) for analysis and archive the other half. Quarter the larger organic fiber bundles the same way, keeping half to proceed to the ashing step and half for archival purposes.

Place the two quarters for ashing into a preweighed crucible. Weigh the split and record the results.

11.2 Ashing

Place the ceramic crucibles containing the samples into a furnace.

The furnace program should proceed as follows:

1. Increase temperature by 1 °C/minute until sample reaches 250 °C.
2. Hold temperature at 250 °C for 4 hours.
3. Increase temperature by 1 °C/minute until sample reaches 480 °C.
4. Hold temperature at 480 °C (sufficient for decomposing organics) for 8 hours. Do not exceed 500 °C.
5. Shut off furnace.
6. Allow sample to cool before removing from furnace.
7. Weigh the ashed sample to the nearest 0.0005 g and record the result.

11.3 Sieving

Sieve the sample through a 150-µm sieve using a Retsch ultrasonic sieve shaker, or similar. Three-inch diameter sieves are recommended to minimize sample loss from particles being trapped in the sieve. The ultrasonic shaker will be operated at 20-minute intervals at the following settings: 20, 40, 60, 70, 80, then back down to 50 and 20. This will provide amplitudes ranging from 0 to 1.5 mm.

Transfer the large and small fractions to clean pieces of weighing paper and weigh to the nearest 0.0005 g. Archive the fraction greater than 150-µm.

11.4 Preparation of Sample for Polarized Light Microscopy

Split the less than 150-µm sample fraction using the cone and quarter method. Collect one corner for analysis and archive the other three quarters. Weigh the quarter split to the nearest 0.0005 g and place it into a glass vial. Make a suspension of 10-20 mg dust per mL of isopropanol. The amount of isopropanol needed will vary depending on the amount of dust; the target dilution is 10-20 mg per mL.

Cut an Eppendorf pipette tip with a razor blade to increase the opening to approximately 1 mm.

Place the suspension in an ultrasonic bath for one minute, then remove the suspension from the ultrasonic bath and shake it gently to suspend all particles. Collect a 10-µL aliquot of the mixture using an Eppendorf pipette with the modified tip and transfer to a glass slide. Prepare 4 such slides. Allow them to dry, then add a drop of 1.55 (or 1.605) refractive index oil.

11.5 Preparation of Sample for SEM Analysis

Prepare the SEM substrate on aluminum stubs using 0.4-µm pore size polycarbonate filters, carbon adhesive tabs. Using an 11 mm filter punch and placing the filter between two filter separators, punch a circle the size of the

carbon tab into the filter. Place carbon adhesive tab affixed to an aluminum stub on the dull side of the 11-mm polycarbonate filter such that the shiny side of the filter exposed. If available, a 13-mm diameter polycarbonate filter may be used in place of the punched out 11-mm filter.

Collect a 10-µL aliquot of the mixture from the PLM sample preparation using the Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate. This will yield a loading on a 12-mm SEM stub of about 100-200 µg, which is a moderately heavy loading. Adjust the number of aliquots as needed to obtain the target loading.

Prepare a 10-fold dilution of the above suspension to get a suspension of 1-2 mg dust per mL of isopropanol. Sonicate the suspension in an ultrasonic bath for one minutes. Remove the suspension and gently shake it to suspend all particles. Wait one minute to allow the coarse particles to settle. Collect a 10-µL aliquot of the suspended mixture using an Eppendorf pipette with the modified tip and transfer to a prepared polycarbonate/adhesive tab substrate. This will yield a loading on a 12-mm SEM stub of about 10-20 µg, which is a light loading. Adjust the number of aliquots as needed to obtain the target loading.

Alternatively, prepare a lightly loaded sample using the filtration method as follows: Use a Millipore filter apparatus for use with 25-mm filters for filtration. Place a few drops of isopropanol on the fritted glass surface and place the 25-mm polycarbonate filter (0.4-um pore size) on the isopropanol. Attach the top of the apparatus and add a few milliliters of isopropanol to the filter so that no part of it is exposed to air. Sonicate the suspension (diluted as described in previous paragraph) in an ultrasonic bath for one minute. Remove the suspension and gently shake it to suspend all particles. Wait one minute to allow the coarse particles to settle. Collect 1 mL of the suspended mixture using a pipette and filter it through the polycarbonate filter. Actual amounts for filtration will vary based on sample loading. The goal is to have a loading on a 12-mm SEM stub of about 10-20 µg, or about 5-10 percent area coverage, which is a light loading. Adjust the volume of the aliquot to filter as needed to obtain the target loading.

Place the filter on a carbon adhesive tab on a standard SEM aluminum mount. The filter needs to be completely flat on the SEM stub. This can be achieved by forming the wet filter into a gentle U-shape using forceps and the side of the forefinger, then placing the bottom curve of the filter onto the center of the carbon adhesive tab and slowly releasing the sides so they lay flat. Trim the edges of the filter using a razor blade.

After drying, coat the samples on the polycarbonate or polycarbonate/adhesive tab substrates with carbon using a carbon evaporator with a rotating stage. Transfer the stubs to the SEM in a clean, covered container.

12.0 Analysis

12.1 Analysis by Polarized Light Microscopy

Polarized light microscopy will be conducted using the general techniques outlined in EPA 600/R93/116 (4). For this procedure, four slides (prepared as described in section 11.4) will be analyzed. The fraction of fibers with refractive index greater than 1.55 (or 1.605) will contain mineral wool, which includes both slag wool and rock wool, and possibly some E-type glass and ceramic fibers. The fraction of fibers with refractive index less than 1.55 (or 1.605) will contain primarily soda-lime glass fibers. For the validation study, numbers of fibers greater than **and** less than 1.55 (1.605) refractive index will be counted. Dispersion staining and becke line techniques may be used. Fiber point counting will be performed at 100 x magnification.

If more than 20 mineral wool fibers are found, continue counting and recording all of the fibers above and below the index oil refractive index. Report both raw fiber counts per refractive index category and number of fibers from each category per gram of sample. Continue on to step 12.2.1 to determine the ratio of slag wool to other fibers with refractive index greater than 1.55 (or 1.605) using EDS as described below.

If less than 20 mineral wool fibers are found on each slide, count the number of slag wool fibers using SEM/EDS and report as number of fibers per gram of sample.

12.2 Analysis by SEM/EDS

12.2.1 Screening for Slag Wool

Operating conditions for the JEOL 6460-LV SEM are 15 kV, 0.5-5-nA beam current, 10-mm working distance (system dependent), and zero degree tilt.

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate into the SEM. Use the backscattered electron mode at 100x magnification to quickly distinguish carbon fibers from inorganic fibers (carbon fibers may be visible, but not as bright in a BEI). Identify all inorganic fibers over 25 µm in length (smaller fibers cannot be reliably detected at the 100x operating magnification). When an inorganic fiber is found, identify the composition of the particle by EDS. Slag wool is the primary fiber of interest. Record all inorganic fiber results as number of fibers for each fiber type.

For the samples with high fiber loading, as determined by PLM as described in section 12.1, count fibers per type until a statistical representation of the ratios of fiber compositions in the sample is achieved. Report the ratio (by fiber number) of slag wool fibers to total MMVF fibers corresponding to the high RI. Use this ratio to correct the total number for high RI fibers counted by PLM to number of slag wool fibers present.

For the samples with low fiber loading, as determined by PLM as described in section 12.1, scan the entire stub to determine the number of fibers per type. Report the slag wool fiber results as the number of slag wool fibers/gram of sample.

12.2.2 EDS Screening for Gypsum/Anhydrite

Place the more concentrated sample deposited directly on the polycarbonate/adhesive tab substrate in the SEM. Choose a random field at 100x magnification and perform an EDS analysis on the entire field. Look for the presence of sulfur in this field. If sulfur is present, continue to Section 12.2.3 or 12.2.5 for analysis of gypsum and concrete by mapping or particle analysis. If it is not present, repeat the analysis on another random field. If sulfur is still not present, mark the sample as non-detect (ND) for sulfur.

12.2.3 X-Ray Mapping for Gypsum

Place a more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM. Collect binary backscattered electron images (particles white and background black, shadow off) and secondary electron images for 10 non-overlapping, random fields at 500 x magnification. Collect x-ray maps for Na, Mg, Al, Si, S, Ca, and Fe at each of these fields. Fields containing MMVF will not be used for this analysis. Operating parameters for the SEM are the same as those for analyzing slag wool. Acquisition parameters for x-ray mapping using the NORAN System Six Software are time constant 14 (mapping mode, 11333 cps), 10-20 % deadtime, 256 x 256 image resolution, 20 second frame time, and 100 frames collected (about 40 minutes total acquisition time). Secondary electron images will be used for reference only. Save all of the maps and electron images in TIFF format.

Open the backscattered electron image and the Ca and S x-ray maps in Adobe Photoshop. Make sure that all of the element maps are the same size and resolution by choosing Image Size from the Image Menu and changing the pixel size or the resolution as needed. The presence of gypsum can be determined by overlapping the Ca and S maps.

Perform the following functions in Adobe PhotoShop. (A macro is in development to perform the following functions to decrease user time and human errors in adjusting the threshold.)

1. Convert each of the three images to grayscale (Image → Mode → Grayscale).
2. Perform an auto contrast and brightness on each image and map to increase the scale of colors (Image → Adjustments → Auto Levels).
3. Threshold each element map, Ca and S (do not analyze the backscattered electron image at this time), by going to the Image Menu and choosing Adjustments → Threshold. Adjust the threshold to 128. The background will be black and the particles white.
4. Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black.
5. Copy the S map and paste it over the Ca map in a separate layer in the file and change the opacity (located in the Layers window) to 50 % for the S map layer. The black areas are gypsum/anhydrite.
6. Display a histogram of the image in expanded mode by selecting the Histogram tab on the Navigator Window (or under the Image Menu in some versions of Photoshop). Place the cursor over the line for the black area and record the percentile for the black area. This is the percentage of particles containing Ca and S in the entire field.

NOTE: If a binary backscattered electron image is obtained during data collection, then steps 7-11 may be deleted. The Invert function will, however, need to be applied to make the particles black and the background white before continuing to step 12.

7. Begin analysis of the backscattered electron image. Select the particles by going to the Select Menu and choosing Color Range. Go to the selection pulldown menu and choose Highlights.
8. Fill the selection with black by going to the Edit Menu → Fill and choosing black from the color pulldown menu.
9. Select the inverse areas by going to the Select Menu and selecting Inverse.
10. Fill the selection with white by going to the Edit Menu → Fill and choosing white from the color pull down menu.
11. Deselect the area by clicking on the image.
12. Perform the Threshold and Histogram functions for the backscattered electron image

as outlined in 3 and 6. Record the histogram result for the backscattered electron image.

Determine the area percent of gypsum by performing the calculations in Section 13.0.

12.2.4 X-Ray Mapping for Ca-Rich Particles

Analysis of components of concrete will be performed on the same fields as the gypsum/anhydrite analysis. At this time, only a method for the determination of the area percent of Ca-rich particles is presented. See Section 2.1 for discussion.

Perform the following steps on the Ca x-ray map Tiff file in Adobe Photoshop:

1. Convert the Ca x-ray map to grayscale (Image → Mode → Grayscale).
2. Perform an auto contrast and brightness on the map to increase the scale of colors (Image → Adjustments → Auto Levels).
3. Threshold the Ca map by going to the Image Menu and choosing Adjustments → Threshold. Adjust the threshold to 128. The background will be black and the particles white.
4. Invert the image (Image→ Adjustments →Invert) to make the background white and the particles black.
5. Display a histogram of the image. Place the cursor over the line for the black area and record the percentile for the black area. This is the area percent coverage of particles containing Ca in the entire field.

Determine the maximum area percent coverage of non-gypsum, Ca-rich particles by performing the calculation in Section 13.0.

12.2.5 Particle Analysis for Identification of Gypsum and Concrete.

Place the more dilute sample, deposited directly on the polycarbonate/adhesive tab substrate or prepared by filtration, in the SEM. Particle analysis will be used to identify gypsum and concrete particles.

Perform particle analysis at 500 x magnification. All other operating parameters for the SEM are the same as those used to analyze for slag wool (Section 12.2.1). A binary backscattered electron image should be used in particle analysis mode. Particle analysis parameters should be set to analyze all particles in the field greater than 0.5 μm and to separate touching particles. For particles greater than 5 μm, scan the entire particle; spot analysis is adequate for smaller particles. The x-ray spectrum and counts for all particles, and an image of particles > 20 μm long, will be recorded and saved. Other particle parameters to be reported will include the maximum, minimum, and average diameters, the aspect ratio, and area of each particle.

It will be necessary to review data collected by automated software to ensure data integrity. An Excel spreadsheet, in conjunction with images and x-ray data, may be used for this purpose. Particles should be sorted into one of three categories: Ca-S (gypsum), Ca-rich, and Other. Aid in identification of particles may by facilitated by referencing the U.S. Geological Survey's WTC Dust Particle Atlas (1). A particle classification protocol will be developed based on the data from the validation study.

The number of particles analyzed will be determined using the results of the validation study. For the study, the area percent of each component should be within 10% relative error or better. Typically, data for 1000 – 1200 particles should be acquired.

Results for particle analysis will be recorded as area percent gypsum and area percent concrete particles for each field and average area percent for the each component in the sample.

13.0 Data Analysis and Calculations

Table 60 To determine the concentration of slag wool in fibers/gram, perform the following calculations:

Determine the number of fibers with RI > 1.55 (or 1.605):

fibers identified ÷ mg of sample on slide × 1000 = fibers/gram on slide

Determine the percentage of fibers with the composition of slag wool with RI > 1.55 (or 1.605):

$$\frac{\text{Fibers/gram on slide} \times \text{\# fibers identified as slag wool}}{\text{Total number of fibers identified by EDS with RI > 1.55 (or 1.605)}} = \text{fibers slag wool/gram on slide}$$

Back calculate to the number of fibers per gram of the original sample:

$$\frac{\text{Fibers slag wool/g on slide} \times \text{g after sieving} \times \text{g sample after ashing}}{\text{g before sieving} \times \text{g sample before ashing}} = \text{Total f/g of sample}$$

Table 61 To determine the area percent of gypsum/anhydrite from the x-ray mapping procedure, perform the following calculations:

Determine the area percent of gypsum/anhydrite in each field of view.

$$\frac{\text{\% of black area in Ca-S map overlay}}{\text{\% of black area in BSE image}} \times 100 = \text{area \% gypsum}$$

Calculate the average percentage of gypsum/anhydrite for the sample.

$$\frac{(\text{area \% gypsum})_{f1} + (\text{area \% gypsum})_{f2} + \ldots}{\text{number of fields}} = \text{Avg. area \% gypsum}$$

Table 62 To determine the maximum area percentage of Ca-rich particles, which includes concrete particles, from the x-ray mapping procedure, perform the following calculations:

Determine the area percent of non-gypsum Ca-rich particles in each field of view:

$$\frac{\text{(\% black area Ca map) – (\% black area Ca-S map)}}{\text{\% black area on BSE image}} = \text{\% non-gypsum Ca-rich particles}$$

Calculate the average percentage of non-gypsum Ca-rich particles for the sample:

$$\frac{(\text{area \% Ca-rich particles})_{f1} + (\text{area \% Ca-rich particles})_{f2} + \ldots}{\text{number of fields}} = \text{Avg. area \% Ca-rich particles}$$

Table 63 Calculate the area percent for gypsum and concrete by summing the areas of each particle in for each particle type and dividing by the total area analyzed:

$$\frac{\text{area gypsum 1 + area gypsum 2 + … x 100}}{\text{total area analyzed}} = \text{area percent gypsum (do likewise for concrete)}$$

Rules for concrete and gypsum classification are currently being developed.

14.0 References

1. Lowers, Heather A., Meeker, Gregory P., Brownfield, Isabelle K., 2005. World Trade Center Dust Particle Atlas: U.S. Geological Survey Open-File Report 2005-1165. On the web at http://pubs.usgs.gov/of/2005/1165/.
2. Meeker, G.P., Taggart, J.E., and Wilson, S.A., 1998. A Basalt Glass Standard for Multiple Microanalytical Techniques. Proceedings: Microscopy and Microanalysis 1998. Microscopy Society of America.
3. A polished and carbon coated calibration reference sample of BIR1-G may be obtained by contacting Stephen Wilson, U.S. Geological Survery, MS 973, Denver Federal Center, Denver, CO, 80225, swilson@usgs.gov.
4. Perkins, R.L. and Harvey, B.W., 1993, TEST METHOD: Method for the Determination of Asbestos in Bulk Building Materials, EPA/600/R-93/116.

15.0 Appendix: DATA SHEETS

Determination of Slag Wool Fibers in Dust- PLM with Dispersion Staining

Sample ID: Project:
Analyst:
Circle One: Original Duplicate Triplicate Date:

General Sample Appearance:_____
Homogeneous?: Y

Structure #	RI Fluid		Dispersion Staining		Becke Line		Fiber			Comments
	1.55	1.605	>RI	<RI	>RI	<RI	MW	non-MW	chrysotile	

SEM Sheet

Reference ASTM - D5755-03

Report Number: _____ **Preparation Date:** _____ **By:** _____
Sample Number: _____ **Analysis Date:** _____ **By:** _____
File Name: _____ **Computer Entry Date:** _____ **By:** _____
Sample Description: _____ **Sample weight:** _____ **grams**
_____ **Dilution Volume:**_____ **mL**
_____ **Volume Aliquot:** _____ **uL**
_____ **Magnification:** _____ **X**

Structure #	Field #	Fiber Type	Length (Microns)	Width (Microns)	Image	EDS

Gypsum and Concrete Report Sheet: X-Ray Mapping Procedure

Sample Number:_____ Analyst:_____ Date:_____

Field #	File Name	% Particles in EDS	% Ca-S area	% Ca-rich area	% Gypsum	% Ca-rich Particles

Averages

APPENDIX E: REPORT FROM THE U.S. EPA CONTRACTOR ON THE SCREENING METHOD STUDY

Versar
6850 Versar Center
Springfield, VA 22151

Ms. Jacky Rosati
US Environmental Protection Agency
E-305-03 109 T.W. Alexander Drive
Research Triangle Park, NC 27711

July 21, 2005

Dear Ms. Rosati:

Attached is a *preliminary* report based on analytical data *thus far received*, for dust samples collected primarily in the New York City area. Most of the samples were taken in areas that, it is believed, were not affected by particulate matter generated during the World Trade Center (WTC) collapse (i.e., background samples). Some of the samples were spiked with one or the other of two dusts that are believed to have originated from the WTC collapse. The analytical protocol was developed by the government, specifically for this project, and was modified as the project developed. The purpose of the testing was to determine if the spiked background dusts could be distinguished from those samples that were not spiked.

Three parameters were measured to make this determination: (1) slag wool fiber content; (2) calcium-rich particle content; and (3) gypsum particle content.

The analytical data indicate that:

- With respect to calcium-rich particles and gypsum particles, spiked samples cannot readily be distinguished from background samples.
- With respect to slag wool content in the samples spiked with the first of the two WTC dusts, spikes at the 10% level may be statistically identifiable as WTC-contamination, although spikes at or below the 5% level are probably not identifiable.
- With respect to slag wool content, samples spiked with 5% and 10% of the second of the two WTC dusts are easily identifiable as WTC-contaminated. Even at the 1% spike level, samples may be statistically identifiable.

The attached *preliminary* report will explain the above conclusions in more detail. However, it must be noted that all of the analytical data from the eight laboratories that performed the analysis has not yet been received. Nevertheless, it is believed that the above conclusions will not likely change once those additional data are incorporated.

Sincerely,

Stephen M. Schwartz, P.E., Q.E.P.
Project Manager

Versar

Preliminary Report
of
Analysis of New York City Area Dust Samples

Purpose:

The objective of this study is to determine if New York City area dusts that are contaminated with varying levels of dusts known to originate from the collapse of the World Trade Center (WTC) can be distinguished from background dusts that are believed not to be contaminated with WTC dusts.

Project Summary:

In the initial portion of the testing, 10 dust samples from New York City areas that are believed not to be contaminated with dusts originating from the collapse of the WTC were used. These are referred to as the first set of *background* samples. An additional background dust sample was spiked at 1, 5, and 10 percent levels (by weight) with dust believed to have originated from the WTC collapse. An additional background sample was spiked at 1, 5, and 10 percent levels with a second dust sample that is believed to have originated from the WTC collapse. Therefore, a set of 16 samples was generated:

- 10 different background dusts
- 3 samples, each consisting of one background dust sample spiked with one source of WTC dust at 1, 5, and 10% levels
- 3 samples, each consisting of one background dust sample spiked with a second source of WTC dust at 1, 5, and 10% levels

Initially, 32 samples were sent to each of eight analytical laboratories (three U.S. government, and five private). The 32 samples consisted of two identical sets (i.e., duplicates) of the 16 samples discussed above. The private laboratories did not know that there were duplicate samples. Further, they did not know which, if any, of the samples contained WTC spikes.

Subsequently, a second set of 28 *different* background samples was analyzed to obtain a better understanding of the variability of background dusts. These 28 samples were sent to only one of the five private laboratories.

It was ultimately agreed that each of the laboratories would perform the following three Scanning Electron Microscopy-based (SEM) analyses on each of the samples they received (see Methodology and Data Analysis section):

- Slag wool fiber content (in number of fibers per gram of dust). Slag wool was a significant component of the WTC insulation material.
- Calcium-rich particle content (in area percent concentration in the SEM field). Such particles are assumed to be indicative of cement/concrete-like particles.
- Gypsum particle content (in area percent concentration in the SEM field). Such particles are assumed to be indicative of "dry wall" (i.e., gypsum-containing wall board).

Conclusions:

A number of conclusions can be drawn from the analytical results thus far obtained. It is not expected that data that are subsequently received will substantially change these conclusions. It must be noted that there are several caveats that affect the quality of the data. Those are discussed later in this report.

2. With respect to calcium-rich particles and gypsum particles, spiked samples cannot readily be distinguished from background samples.

Tables 1 and 2 present the analytical data *thus far available* for calcium-rich and gypsum content respectively. Analysis was performed using SEM and x-ray mapping (XRM) techniques. The shaded areas represent the samples spiked with 1, 5, and 10% WTC dust. The others areas are background samples. Sample designations followed by "(1)" and "(2)" are duplicate samples. (Samples received by the laboratories had random identification numbers, so that the laboratories did not know if any samples were duplicates, nor did they know if any samples contained WTC dust.) In addition, Table 3 is the analysis of a subsequent 28 background samples, analyzed by only laboratory "B". Analysis of calcium-rich and gypsum particles for this sample set is shown on Table 3.

The average of all background samples (including the second set of 28 samples) for calcium-rich particles is 22.3 area percent, with a high value of 66.5% and a low value of 4.2%. The average for the spiked samples is 20.7%, with the highest value being 25.9%. The 1, 5, and 10% spiked samples do not show any trend with respect to calcium-rich particle content (i.e., they do not show any increase as the spike level increases).

The average of all background samples (including the second set of 28 samples) for gypsum particles is 11.7 area percent, with a high value of 56.5% and a low value of 0.1%. The average for the spiked samples is 9.3%, with the highest value being 32.8%. The 1,5, and 10% spiked samples do not show any trend with respect to gypsum particle content.

3. With respect to slag wool content in the samples spiked with the first of the two WTC dusts, spikes at the 10% level may be statistically identifiable as WTC-contamination, although spikes at or below the 5% level are probably not identifiable.

Table 4 presents all the analytical data thus far available for SEM slag wool fiber analysis (as the number of slag wool fibers per gram of dust). The shaded areas represent samples that are spiked at the 1, 5, and 10% levels with WTC dust. Table 3 also presents additional slag wool fiber background-only sample data (next to last column). It can be seen from Figure 1 that for those spiked samples designated as "DB" that at the 5% spike level, the slag wool concentrations probably do not exceed one standard deviation above the average slag wool background concentration (including the Table 3

background data). However, at the 10% spike level, the slag wool concentration typically exceeds one standard deviation (see Figure 2), but never exceeds two standard deviations above the average background sample concentration. The average background concentration is about 27,400 fibers per gram. The standard deviation is about 40,100 fibers per gram.[1]

It should be noted that there is a trend showing a clear increase in slag wool fiber concentration from the 1% to the 10% spike level (see "DB" sample shaded area on Table 4). However, the numerical values of those concentrations, as noted above, are still less than two standard deviations above the average concentration.

4. With respect to slag wool content, samples spiked with 5% and 10% of the second of the two WTC dusts are easily identifiable as WTC-contaminated. Even at the 1% spike level, samples may be statistically identifiable.

The slag wool content data for the samples spiked with the WTC dust shown in Table 4 as "USGS" are easily identifiable. As can be seen in Figures 4 and 5, samples spiked with the USGS WTC dust at the 5 and 10% levels are essentially all more than two standard deviations above the average background sample concentration. (Average plus two standard deviations would be about 108,000 fibers per gram.[2]) At the 1% spike level though, WTC dust is more difficult to identify because the slag wool concentrations are mostly between one and two standard deviations above the average background sample (see Figure 3).

5. With respect to slag wool content, clearly, there is a large difference between the two WTC dust spikes used. In the "DB"-spiked samples, as noted above, it is expected to be more difficult to determine a significant slag wool fiber concentration difference from background. The "USGS"-spiked samples clearly had significantly more slag wool fiber content than the "DB" samples.

6. Examining Tables 1, 2, and 4 and the Figures, it can be seen that the analyses for the duplicate samples rarely replicate one another. However, the variation between duplicate sample values (i.e., int***ra***lab) is about half of the variation between individual laboratory values (int***er***lab).[3]

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[1] Background concentration data for this analysis excluded several samples that were known to have high slag wool content, specifically the C1-RTP samples (see Table 4), and samples C2,3,4,5,6 (see Table 3).
[2] Ibid.
[3] For slag wool fiber analysis, the average difference between the analyses of duplicates (i.e., int***ra***lab differences) is about 50% of one standard deviation of the between-laboratories analyses (i.e., int***er***lab differences). For both calcium-rich and gypsum particle analysis the average intralab difference is 20% of the interlab difference.

Methodology and Data Analysis:

The analytical protocol was developed specifically for this project by one of the government laboratories, and modified by all laboratory participants at a meeting held for that purpose. All laboratory participants held weekly conference calls as the analytical program was proceeding to discuss general issues with the protocol. Additional modifications were made to the protocol based on those conference calls.

The original protocol included analysis by Polarized Light Microscopy (PLM), so data are also available for PLM analysis. The PLM analyses were curtailed because it became obvious that PLM could not adequately differentiate between fiber types. Further, total fiber concentrations were also determined, both by PLM and SEM methods, but those data are not presented in this report.

Caveats:

There are a few factors that may contribute to data uncertainty. Nevertheless, it is unlikely that these factors will alter the above major conclusions. Some of these factors are as follows:

1. As noted earlier, not all of the analytical data have been received.
2. Dust samples were collected by several methods. Evaluation of the sampling methodology was not part of the study.
3. To determine fiber concentration, fibers were counted using an SEM. Different laboratories diluted samples to different levels before counting, introducing some variability of results.
4. Laboratory equipment capabilities and personnel skills varied.

TABLE 1: SEM X-Ray Mapping – Calcium-Rich Area Percent

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)		23.4		20.4	14.4	11.6	30.7	45.8	Chittenden Avenue, Manhattan
AP5(2)		22.4		22.1	16.8	9.8	39.6	48.9	
CMC(1)		27.7		21.8	6.7	7.9	55.1	60.4	Columbia Medical Center, W 68th Street, Manhattan
CMC(2)		34.1		21.5	20.4	10.1	38.9	55.2	
HS3(1)		10.3		14.1	6.4	15.3	29.1	63.1	Teaneck, NJ
HS3(2)		17.8		13.3	14.8	6.5	44.0	49.7	
WGS(1)		22.8		13.2	13.9	7.7	58.4	53.4	Nassau County, LI
WGS(2)		19.9		16.3	5.7	7.4		52.3	
MW(1)		12.2		14.2	12.6	7.6	49.3	46.3	West End Ave Between 105th and 106th Streets, Manhattan
MW(2)		12.0		10.9	8.3	5.7		49.8	
DB1%(1)		18.2	15.6	14.0	18.3	8.9	55.9	50.2	4 Albany Street Spiked into NE Queens background dust
DB1%(2)		13.1		14.1	15.4	9.4		52.2	4 Albany Street Spiked into NE Queens background dust
DB5%(1)		13.4	23.0	16.1	10.8	9.0	40.0	49.0	4 Albany Street Spiked into NE Queens background dust
DB5%(2)		20.5		12.9	4.1	7.5		39.6	4 Albany Street Spiked into NE Queens background dust
DB10%(1)		14.4		15.2	8.6	8.0	50.7	40.6	4 Albany Street Spiked into NE Queens background dust
DB10%(2)		12.6	18.9	14.9	10.8	8.1		48.8	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)		13.2		11.3	7.5	6.1	57.1	66.5	Research Triangle Park, NC
C1-RTP(2)		16.2		11.9	5.6	4.2		61.0	Research Triangle Park, NC
USGS1%(1)		16.5		17.4	12.4	7.6		43.2	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		21.0	26.6	11.1	5.7	7.4		41.9	USGS Dust Spiked into NE Queens background dust
USGS5%(1)		14.8		14.2	11.9	6.7		53.6	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		14.6	26.4	16.2	10.7	8.3		51.8	USGS Dust Spiked into NE Queens background dust
USGS10%(1)		17.0		15.8	10.9	8.9		40.0	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		17.9		12.1	9.8	8.3		45.1	USGS Dust Spiked into NE Queens background dust
USC(1)		12.3		11.1	19.5	5.4	43.5	46.1	Federal Courthouse, White Plains, NY
USC(2)		9.4		9.5	6.6	7.1		40.3	
FP(1)		13.0		11.6	5.9	6.2	42.4	70.9	Federal Courthouse, Central Islip, LI

FP(2)		10.5		10.3	10.3	8.2		61.5	
MUNYC1(1)		25.4		17.1	15.2	6.3	55.6	39.6	Northern Manhattan, Above 70th Street
MUNYC1(2)		19.7		14.0	31.9	8.0		36.6	
MUNYC2(1)		20.4		19.9	27.7	8.9	45.4	56.8	Northern Manhattan, Above 70th Street
MUNYC2(2)		17.6		14.4	13.6	7.3		57.8	

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples.

TABLE 2: SEM X-Ray Mapping - Gypsum Area Percent

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)		8.0		14.4	0.9	2.5	34.1	26.1	Chittenden Avenue, Manhattan
AP5(2)		20.3		11.3	1.8	1.6	31.3	33.7	
CMC(1)		4.3		4.8	0.2	1.1	26.1	22.4	Columbia Medical Center, W 68th Street, Manhattan
CMC(2)		6.9		3.0	1.0	1.0	30.8	17.6	
HS3(1)		5.9		9.2	0.3	5.7	44.0	42.9	Teaneck, NJ
HS3(2)		14.9		11.0	2.3	1.5	29.0	40.5	
WGS(1)		2.9		5.4	0.2	0.4	19.0	42.2	Nassau County, LI
WGS(2)		6.1		4.7	0.2	0.3		39.1	
MW(1)		3.8		7.0	0.2	0.7	23.2	37.6	West End Ave Between 105th and 106th Streets, Manhattan
MW(2)		5.4		5.3	0.1	1.1		41.6	
DB1%(1)		7.2	13.8	5.7	3.0	0.6	22.0	28.0	4 Albany Street Spiked into NE Queens background dust
DB1%(2)		7.1		5.2	1.1	1.3		30.0	4 Albany Street Spiked into NE Queens background dust
DB5%(1)		7.3	3.4	5.5	0.7	1.2	29.1	24.3	4 Albany Street Spiked into NE Queens background dust
DB5%(2)		6.1		5.5	0.1	1.6		28.9	4 Albany Street Spiked into NE Queens background dust
DB10%(1)		6.5		7.8	0.5	1.0	25.7	27.0	4 Albany Street Spiked into NE Queens background dust
DB10%(2)		5.0	8.7	4.8	0.6	1.9		28.5	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)		8.5		9.7	0.2	1.3	24.5	53.4	Research Triangle Park, NC
C1-RTP(2)		8.7		8.2	0.3	0.8		50.4	Research Triangle Park, NC
USGS1%(1)		6.3		5.8	1.0	0.9		29.4	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		5.4	15.2	4.1	0.2	0.9		29.2	USGS Dust Spiked into NE Queens background dust
USGS5%(1)		7.7		5.7	0.9	1.1		29.3	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		2.5	9.8	4.1	0.5	2.4		21.7	USGS Dust Spiked into NE Queens background dust
USGS10%(1)		6.3		7.1	1.2	1.1		30.9	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		4.8		4.8	0.7	1.4		32.8	USGS Dust Spiked into NE Queens background dust
USC(1)		4.8		5.2	1.2	0.7	24.9	27.1	Federal Courthouse, White Plains, NY
USC(2)		6.2		4.2	0.2	2.4		32.4	

FP(1)		11.6		5.4	0.3	1.2	24.5	56.5	Federal Courthouse, Central Islip, LI
FP(2)		4.4		6.1	0.6	1.5		40.0	
MUNYC1(1)		10.5		9.2	1.2	0.9	26.8	24.1	Northern Manhattan, Above 70th Street
MUNYC1(2)		3.0		5.5	1.4	1.0		26.3	
MUNYC2(1)		5.5		6.1	9.2	2.5	31.0	30.8	Northern Manhattan, Above 70th Street
MUNYC2(2)		4.2		6.0	0.7	1.8		29.5	

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples.

Table 3: New York City Background Dust Samples

Sample No.	EPA Sample ID	SEM XRM		SEM (Heavy Loading)		Particle Count		
		Calcium-Rich (area %)	Gypsum (area %)	Slag Wool (fibers/g)*	Total Fibers (fibers/g)	Slag Wool	Total Fibers	
1	HS1-06-01**	5.8	5.8	35,565	104,603	9	25	Stony Brook, LI
2	HS1-06-02	16.3	4.0	230,769	523,077	15	34	
3	AP2-07-01	12.4	8.5	32,432	113,514	6	21	West End Ave between 72nd and 73rd Streets
4	AP2-07-02	9.9	7.2	7,692	130,769	2	34	
5	AP3-08-01	10.5	4.7	12,500	212,500	2	34	30th Ave between 21st and 23rd, Queens
6	AP3-08-02	17.3	7.6	<3,636	21,818	0	6	
7	HS2-09-01	25.6	9.6	7,605	22,814	2	6	70th Street between 20th and 21st Ave, Brooklyn
8	AP4-10-01	13.1	11.5	42,857	485,714	3	34	79th St between York and East End Ave, Manhattan
9	AP7-14-01	17.5	6.0	3,333	23,333	1	7	92nd Street between Columbus and CPW, Manhattan
10	CMC-17-01	30.5	10.4	4,651	23,256	1	5	Columbia Medical Center, W. 168th St, Manhattan
11	HS3-18-01	14.3	9.0	11,858	71,146	3	18	Teaneck, NJ
12	WGS6557	10.2	4.2	34,826	44,776	7	9	Nassau County, LI
13	WGS5826-1	18.1	6.2	15,564	54,475	4	14	Nassau County LI
14	PT152W88	17.5	8.4	17,021	46,809	4	11	88th Street between Amsterdam and Columbia, Manhattan
15	PT152W88-2ndFl	15.8	5.6	19,305	42,471	5	11	88th Street between Amsterdam and Columbia, Manhattan
16	CY321W80	14.7	12.2	30,888	34,749	8	9	80th Street between Riverside and East End Ave, Manhattan
17	MW924WEAve	21.1	10.1	8,097	28,340	2	7	West End Ave between 105th and 106th Streets
18	C2**	7.9	6.1	46,703	102,890	11	24	Research Triangle Park, NC
19	C3	16.8	3.4	170,309	321,696	18	34	Research Triangle Park, NC
20	C4	13.7	3.8	160,772	227,760	24	34	Research Triangle Park, NC
21	C4 (no date)	17.5	8.8	488,372	790,698	21	34	Research Triangle Park, NC
22	C5	16.7	7.2	74,236	148,472	17	34	Research Triangle Park, NC
23	C6	10.0	10.9	280,762	415,039	23	34	Research Triangle Park, NC
24	N-01S	12.3	7.8	369,231	523,077	24	34	Edison, NJ

25	Nevins Ct	16.0	9.6	<4,367	91,703	0	21	Edison, NJ
26	E Curtis Ave**	7.9	9.0	5,173	24,138	2	7	Edison, NJ
27	LBI	7.3	16.3	<3,636	61,818	0	17	Long Beach Island, NJ
28	Mixture	19.1	11.8	7,194	35,971	2	10	NE Queens
Average		**14.9**	**8.1**	**84,709**	**168,837**			
Standard Deviation		**5.5**	**3.0**	**128,759**	**200,808**			
Coeff. Of Variance		**0.4**	**0.4**	**1.5**	**1.2**			

***** A fiber count of one fiber was used to calculate the analytical sensitivity for non-detects.

****** Internal laboratory duplicates were run on these samples. The result shown is the average of the two duplicates ("<" samples were assumed to be 0).

TABLE 4: SEM - Slag Wool Fiber Count/Gram of Sample

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)	non-det.	3,663		non-det	<249	<500	2,470	<7,386	Chittenden Avenue, Manhattan
AP5(2)		<3636		6,980	<667	500	13,910	<7,698	
CMC(1)	non-det.	3,448		11,800	<282	<4,500	5,780	<7,241	Columbia Medical Center, W 68th Street, Manhattan
CMC(2)		<3875		9,620	309	667	6,100	<6,289	
HS3(1)	16,393	7,299		19,000	<286	2,750	<6,320	<7,576	Teaneck, NJ
HS3(2)		7,692		18,600	<667	5,060	7,370	34,813	
WGS(1)	5,900	34,221		26,400	<256	1,630	9,480	16,077	Nassau County, LI
WGS(2)		10,753		18,100	6,990	<30,500	3,520	18,399	
MW(1)	12,232	18,939		18,700	1,320	1,000	13,630	17,301	West End Ave Between 105th and 106th Streets, Manhattan
MW(2)		3,717		31,800	893	<45,500	18,080	<9,497	
DB1%(1)	5,747	10,909	5,451	29,900	<2,000	1,920	7,650	15,924	4 Albany Street Spiked into NE Queens background dust
DB1%(2)	34,826	17,422	9,133	27,300	3,770	12,500	1,320	16,038	4 Albany Street Spiked into NE Queens background dust
DB5%(1)	72,562	29,197	32,385	50,800	31,000	1,700	6,230	107,143	4 Albany Street Spiked into NE Queens background dust
DB5%(2)	67,797	25,271	33,646	35,800	6,900	14,700	13,040	70,472	4 Albany Street Spiked into NE Queens background dust
DB10%(1)	104,575	66,421	74,837	113,000	108,000	7,000	12,900	114,638	4 Albany Street Spiked into NE Queens background dust
DB10%(2)	84,746	77,778	57,644	95,100	20,400	34,100	25,210	96,696	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)	246,914	159,011		269,000	168,000	38,000	84,650	188,088	Research Triangle Park, NC
C1-RTP(2)		173,585		165,000	21,900	160,000	39,930	318,143	Research Triangle Park, NC
USGS1%(1)	98,039	109,091	50,293	119,000	366,000	79,800	9,200	90,992	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		83,032	50,160	104,000	18,700	79,500	25,370	137,363	USGS Dust Spiked into NE Queens background dust
USGS5%(1)	600,000	404,332		681,000	227,900	433,000	66,450	672,926	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		343,284	364,813	146,000	191,000	197,000	73,330	347,904	USGS Dust Spiked into NE Queens background dust
USGS10%(1)	1,218,855	840,231	531,277	1,620,000	1,410,000	629,000	144,120	734,767	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		1,366,470	521,212	238,000	271,000	372,000	33,040	413,153	USGS Dust Spiked into NE Queens background dust
USC(1)	73,394	56,025		91,800	33,700	15,600	<3,230	29,268	Federal Courthouse, White Plains, NY
USC(2)		41,199		40,700	7,890	48,400	3,540	74,212	
FP(1)	18,519	18,051		16,300	1,100	12,400	11,920	28,249	Federal Courthouse, Central Islip, LI
FP(2)		16,470		31,800	3,920	30,500	<1,181	25,489	

MUNYC1(1)	10,840	7,220		14,400	14,900	13,100	<2,545	6,803	
MUNYC1(2)		3,745		20,200	1,960	<22,300	<1,228	41,118	Northern Manhattan, Above 70th Street
MUNYC2(1)	41,298	28,777		66,500	1,390	17,800	<12,453	123,106	
MUNYC2(2)		48,507		45,500	24,200	30,500	2,330	59,473	Northern Manhattan, Above 70th Street

Samples spiked with WTC dust, at 1, 5, and 10% levels are shaded. All others are background samples.

For data analysis purposes:

- Non-det = Non-detect – zero slag wool fibers were noted in the sample.
- <# indicates that the value was less than the detection limit of the respective laboratory. When this result was reached, the value was divided by the square root of 2.

Table 5: SEM - Slag Wool Fiber Count

Sample Designations	Laboratory Letter Codes								Location Key:
	A	B	C	D	E	F	G	H	
AP5(1)	0	1		0	0	0	2	0	Chittenden Avenue, Manhattan
AP5(2)		0		3	0	1	6	0	
CMC(1)	0	1		5	0	0	4	0	
CMC(2)		0		4	1	0	5	0	
HS3(1)	3	2		8	0	1	0	0	Teaneck, NJ
HS3(2)		2		8	0	3	4	4	
WGS(1)	1	9		11	1	1	6	2	Nassau County, LI
WGS(2)		3		8	7	0	3	2	
MW(1)	2	5		8	6	1	6	2	
MW(2)		6		14	2	0	22	0	
DB1%(1)	1	3	1	13	0	1	7	2	4 Albany Street Spiked into NE Queens background dust
DB1%(2)	7	5	2	12	4	2	1	2	4 Albany Street Spiked into NE Queens background dust
DB5%(1)	8	8	7	22	7	1	6	6	4 Albany Street Spiked into NE Queens background dust
DB5%(2)	12	7	7	16	8	2	11	10	4 Albany Street Spiked into NE Queens background dust
DB10%(1)	16	18	12	48	13	2	10	13	4 Albany Street Spiked into NE Queens background dust
DB10%(2)	15	21	12	42	9	3	25	12	4 Albany Street Spiked into NE Queens background dust
C1-RTP(1)	20	45		116	16	4	22	24	Research Triangle Park, NC
C1-RTP(2)		46		72	30	4	17	37	Research Triangle Park, NC
USGS1%(1)	15	30	9	54	27	11	11	10	USGS Dust Spiked into NE Queens background dust
USGS1%(2)		23	11	47	23	4	22	15	USGS Dust Spiked into NE Queens background dust
USGS5%(1)	99	112		194	25	>20	64	43	USGS Dust Spiked into NE Queens background dust
USGS5%(2)		92	62	65	21	19	27	39	USGS Dust Spiked into NE Queens background dust

USGS10%(1)	181	45	124	450	38	19	18	41	USGS Dust Spiked into NE Queens background dust
USGS10%(2)		45	129	105	19	16	9	49	USGS Dust Spiked into NE Queens background dust
USC(1)	6	13		39	6	13	0	3	
USC(2)		11		18	6	4	2	8	
FP(1)	3	5		7	3	2	2	3	
FP(2)		5		14	4	1	0	3	
MUNYC1(1)	1	2		6	4	3	0	1	
MUNYC1(2)		1		9	2	0	0	5	
MUNYC2(1)	7	8		28	3	3	0	13	
MUNYC2(2)		13		20	24	3	1	7	

Samples spiked with WTC dust, at 1, 5, and 10% levels are highlighted in yellow.

ADDENDUM TO VERSAR REPORT: SEM CALIBRATION DATA

MEMORANDUM

TO: Jacky Rosati

CC: David Friedman

FROM: Stephen Schwartz

DATE: August 5, 2005

SUBJECT: BIR-1G Sample Analyses

Identical mounted and polished reference samples, each designated BIR-1G, were sent to each of the five private laboratories participating in the analyses of dust samples from New York City and elsewhere. The samples were analyzed by Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry (SEM/EDX) to determine their elemental content. The purpose of the study was to determine the variation within and between each of the laboratories, and to assess their ability to identify elements using this technology.

Each of the five laboratories analyzed their BIR-1G sample between 4 and 11 times, as convenient (there was no requirement for a specific number of analyses). The average elemental concentration data for each laboratory is presented in the attached table. For calcium (Ca), magnesium (Mg), silicon (Si), and oxygen (O)[4], which constitute over 80% by weight of the elemental composition, the standard deviation *within* each laboratory, for each element, was typically much less than 10% (i.e., the coefficient of variation). Likewise, the coefficient of variation *between* laboratories for Ca, Mg, Si, and O, as shown on the attached table, was also much less than a 10%. (The graphic presentations of the EDX spectra within and between laboratories also appear to be extremely similar.

Therefore, it can be concluded that each of the laboratories was easily able to achieve excellent precision, by SEM/EDX, in quantifying the elements that were present in larger concentrations.

[4] Some of the laboratories reported results as the weight percent of the elemental oxides, specifically: Na_2O, MgO, Al_2O_3, SiO_2, CaO, TiO_2, FeO, K_2O, and MnO_2. Oxide values were converted to individual elemental values (e.g., Al_2O_3 is about 53% Aluminum, and 47% oxygen by weight).

AVERAGE ELEMENTAL CONCENTRATION REPORTED FOR BIR-1G SAMPLES (Weight Percent of Sample)

Lab	Sodium	Magnesium	Aluminum	Silicon	Calcium	Titanium	Iron	Potassium	Manganese	Oxygen
A										
B										
C										
D										
E	0.44	5.02	8.11	21.60	9.66	0.68	9.68	NR	NR	44.84
F										
G	1.14	5.82	8.75	24.76	7.68	0.52	5.56	0.00	0.00	45.78
H	1.84	5.10	7.88	22.20	10.30	0.64	8.37	0.03	0.21	43.43
AVERAGES	1.14	5.31	8.25	22.85	9.21	0.61	7.87	0.02	0.11	44.68
Standard Dev. (% of Average)	61.40	8.33	5.48	7.35	14.83	13.58	26.73	141.42	141.42	2.65

NR - Not Reported

APPENDIX F: STATISTICAL ANALYSIS AND INTERPRETATION OF TEST RESULTS – LABORATORY QUALIFICATION

Slag wool fiber content as a discriminator for residual WTC contamination in indoor dust sample: Interpretation of multi-laboratory test results

Introduction

Eight laboratories were each challenged with a number of blinded dust sample aliquots to determine number of slag wool fibers per gram. These samples included background dusts from various locations in NYC and a series of samples of common household dust spiked with different levels of WTC collapse dust collected in 2001 or in 2004. The purpose of this endeavor was to assess whether or not the method developed can be used by qualified laboratories to discriminate between WTC and non-WTC impacted dust samples.

In the following discussion, individual laboratories are evaluated and ranked for validity and precision, and then the top performers are further evaluated as groups to determine the expected confidence level for the slag wool content of any individual and randomly assigned)sample.

Laboratory Qualification

Validity: Assessment of validity was conducted by analysis of a series of spiked samples where the expected response ratios are known. The challenge samples consisted of a large volume of non-impacted background dust collected in 2004 from locations in Northeast Queens over ten miles from the WTC site. This dust was subsequently spiked with 1, 5, and 10% WTC dusts by weight using either bulk collapse dusts collected in September 2001 immediately following the disaster (designated as USGS dust), or nominally undisturbed dusts collected in 2004 in the abandoned Deutsche Bank (DB) complex that borders the south side of the WTC complex (designated as 4 Albany dust).

Using units of (# slagwool fibers)/(gram of dust), preliminary analyses showed a mean value of 12,200,000 (s.d. 1,697,056) for USGS dust, 579,667 (s.d. 173,782) for 4 ALBANY dust, and a nominal background level of 7,190. Based on these data, the expected values of slope expressed as [(# slagwool fibers)/(gram dust)]/[% spike level] are 121,928 and 5,725, respectively for USGS and 4 Albany spikes. Specifically, each lab was furnished two samples each of 1, 5, 10% spikes from both 4 Albany and USGS series for a total of 12 spiked samples plus a series of 20 additional background samples collected from random locations all over the greater NYC area.

Scatterplots and linear least squares regressions were constructed for each lab and for each of the two spike series. Preliminary inspection showed no apparent violations of underlying assumptions required for regression analysis (primarily homogeneity of variance); as such no lognormal transformation was performed. Using a forward selection strategy, it was found that a higher order polynomial model does not statistically improve the linear fit; this is expected as the sample set is designed as a linear progression. Also, a simpler but more general “runs” test for each linear regression confirmed these results. Data handling and manipulation was performed with Microsoft Excel SP-2; statistical analyses were performed with SAS 9.1.3 XP-Pro (proc rsreg/lackfit, proc reg, proc

mixed, and proc univariate); graphing, ANOVA, and various other statistical results were performed or verified with GraphPad Prism 3.03. The linear regression results are given in Table 1; a summary of SAS proc rsreg/lackfit results are given in Table 2.

Table 1: Summary of linear least squares regression results.

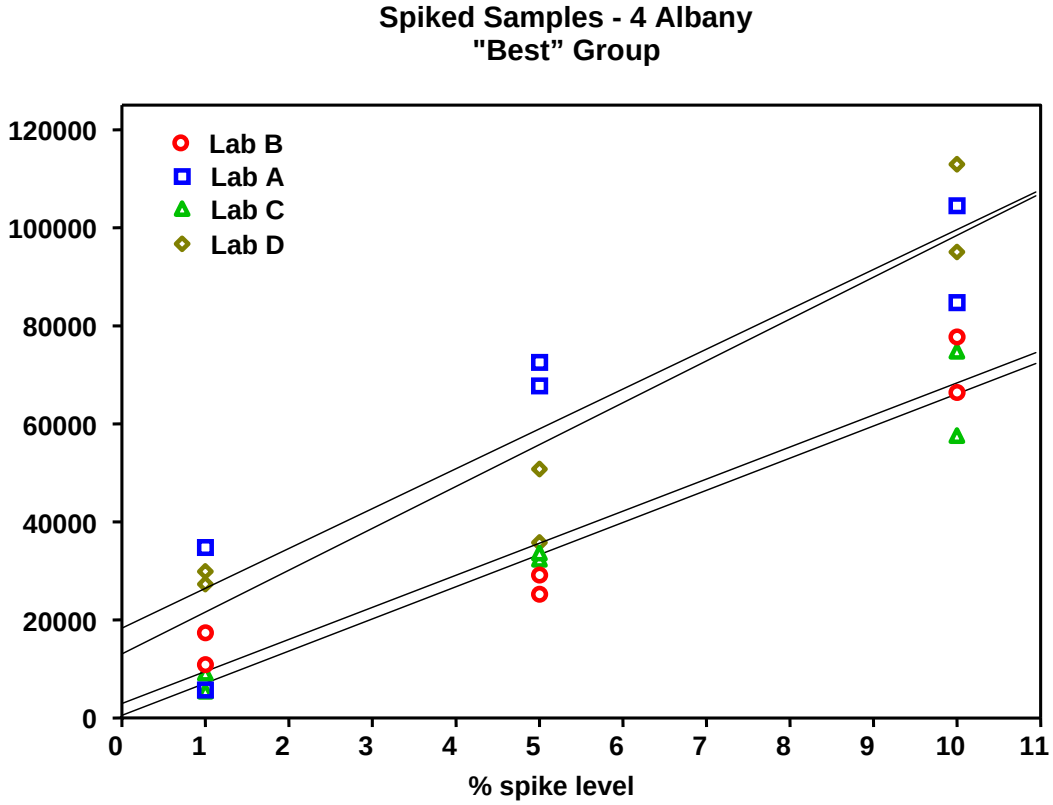
Lab	DB spikes (4 Albany)				USGS spikes (2001 dusts)				
	slope	95% CI (+/-)	sig slope p-value	r2	slope	95% CI (+/-)	sig slope p-value	r2	
A	8126	1760	0.0099	0.8421	124500	488	0.0025	1.0000	note: n =3
B	6541	971	0.0025	0.9190	113300	23460	0.0085	0.8537	
C	6554	691	0.0007	0.9573	52380	5914	0.0030	0.9632	
D	8538	1515	0.0049	0.8881	91340	58230	0.1918	0.3608	
E	6936	3632	0.1288	0.4769	74240	49810	0.2104	0.3571	
F	1523	1288	0.3027	0.2588	46370	14020	0.0298	0.7321	
G	1630	610	0.0559	0.6404	7750	4607	0.1678	0.4144	
H	9695	2645	0.0215	0.7705	49510	21790	0.0855	0.5634	

Table 2. Summary of "lack of fit" tests

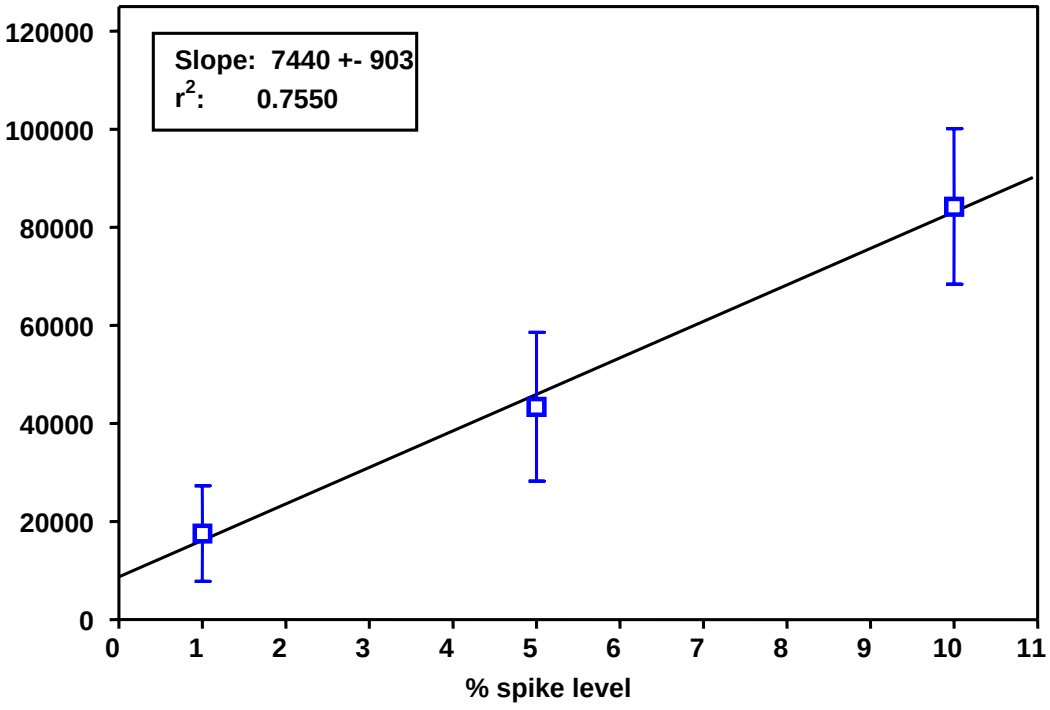
Lab	r2 linear	r2 quad	p-value linear	pvalue quad
A	0.8421	0.0590	0.0149	0.2732
B	0.9190	0.0564	0.0018	0.0791
C	0.9573	0.0001	0.0038	0.9441
D	0.8881	0.0706	0.0040	0.1087
E	0.4769	0.0197	0.1904	0.7547
F	0.2588	0.0445	0.3386	0.6913
G	0.6404	0.0069	0.1018	0.8240
H	0.7705	0.1455	0.0135	0.1070
A,B,C,and D	0.7550	0.0032	0.0001	0.6038
A,B,C,D and H	0.7027	0.0023	0.0001	0.6473

Based on these summaries the linear model is appropriate. Laboratories A and B demonstrate excellent performance across the board: each has r^2 values > 0.80, significant positive slopes with $p < 0.05$, and slopes with the expected magnitude. We caution that Lab A only has three points for the USGS spike results (yellow highlights, Table 1). Fields highlighted in blue indicate potential problem areas. If only the 4 Albany spike series are considered, then Labs C and D can be added to the preferred performer group. This is reasonable because the range covered here is more likely to reflect the range of concern for unknown samples. Although the Lab H results demonstrate a lower r^2 value and a larger 95% CI for slope, this is caused by a single outlying point. As such, there is no reason to exclude Lab H from the analysis. Because Labs E, F, and G fail in more than one category in both spiked data sets, they are not included in the remaining study analysis.

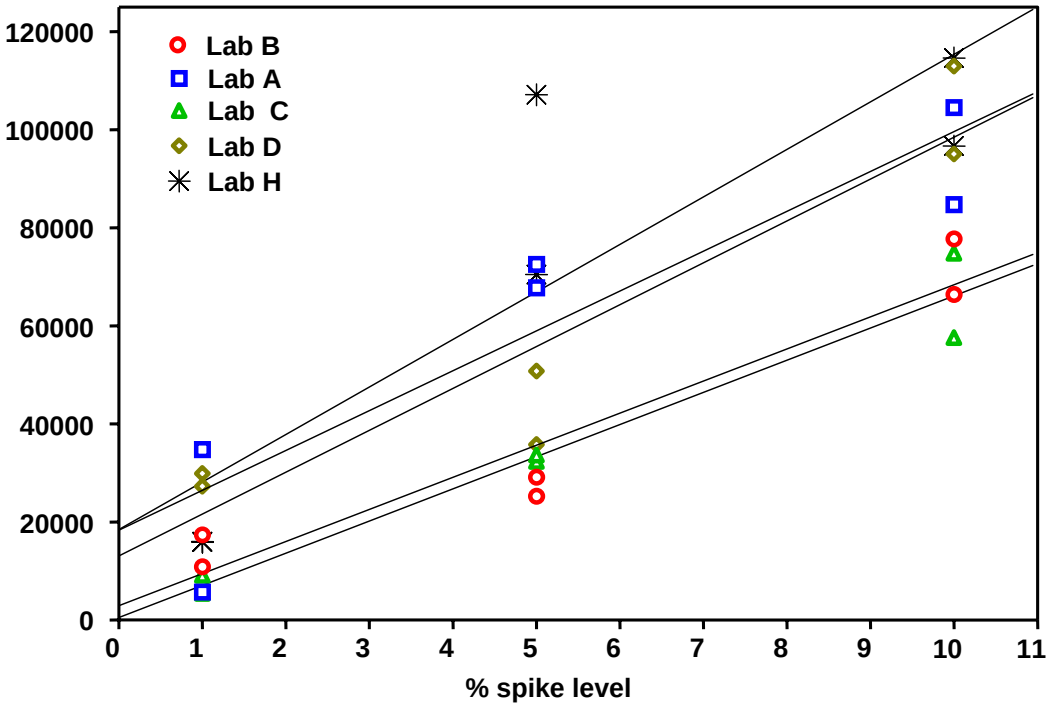
From the above discussion, we can construct two groups of laboratories based on the estimated validity of their results: “Best”, consisting of Labs A, B, C, and D and “very good” consisting of the best group plus Lab H. In the following series of figures, the individual and composite linear regression results for the groups are demonstrated graphically.



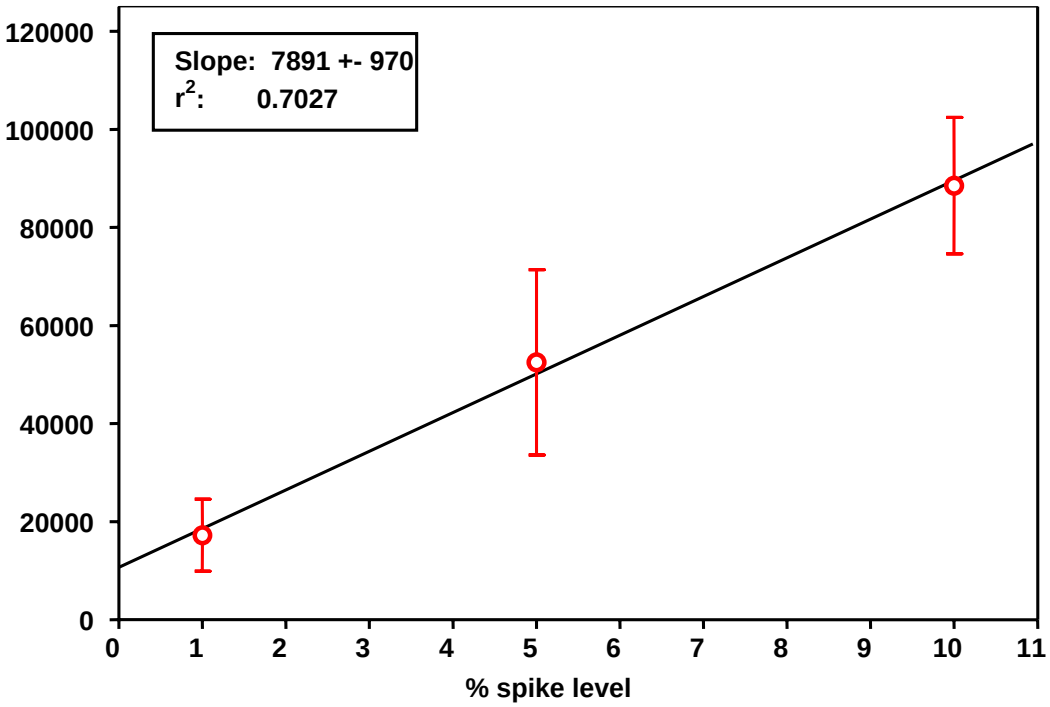
Spiked Samples - 4 Albany
"Best" Group Combined
(Labs A, B, C, and D)



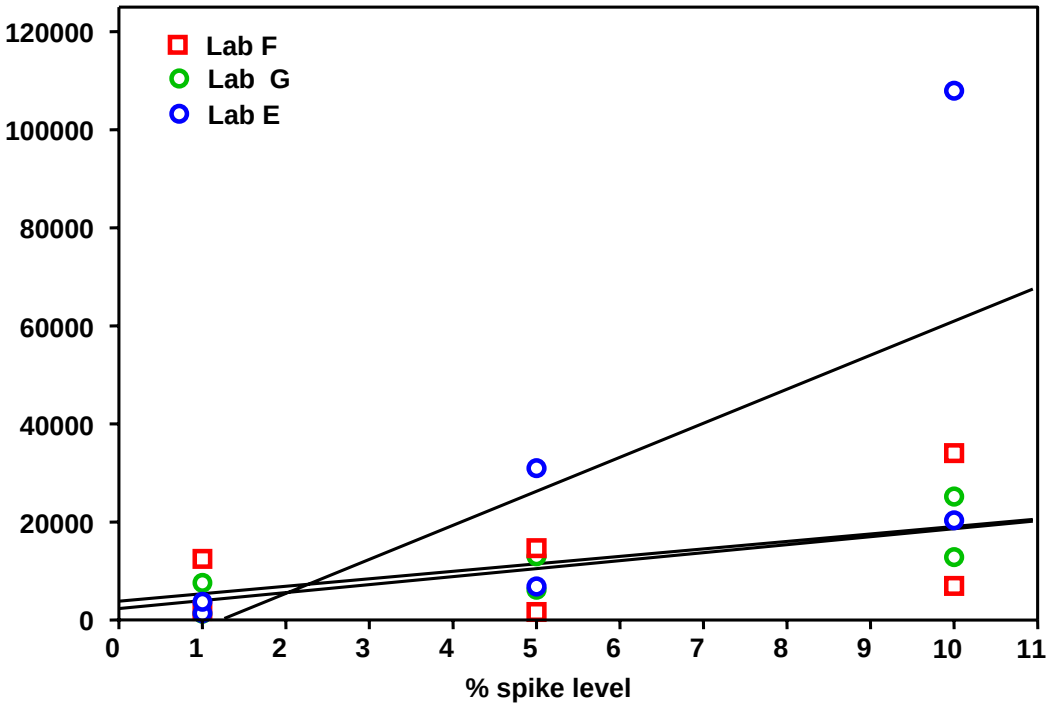
Spiked samples - 4 Albany
"Very Good" Group
(Labs A, B, C, D and H)

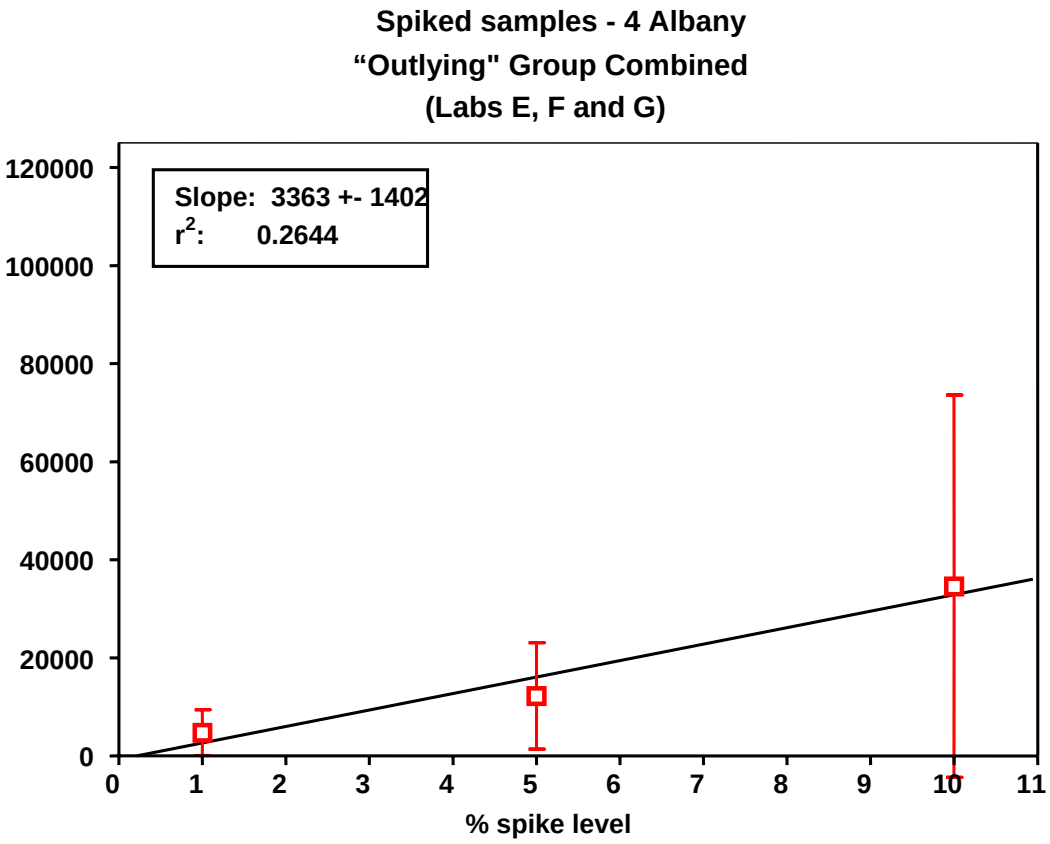


Spiked samples - 4 Albany
"Very Good" Group Combined
(Labs A, B, C, D and H)



Spiked samples - 4 Albany
"Outlying" Group
(Labs E, F and G)





Precision: Up until this point, validity has been assessed only with those samples for which there is some prior knowledge of content. For assessing precision, however, one can use all of the samples (including unknowns) because each laboratory received aliquots of the same set of 32 samples. Furthermore, the sample structure is such that these 32 samples are comprised of 16 paired samples allowing within laboratory precision estimates as well. Although there are a number of statistical options for proceeding, an analysis of variance (ANOVA) and intra-class correlation coefficients (ICC) are pragmatic for these circumstances as samples and laboratories are used in groups. Preliminary analyses of "within" and "among" laboratory results indicate that the underlying distributions (considering all 32 sample results) are not normal based on the Shapiro-Wilk (S-W) test, and that natural log transformation of the data should used to perform analysis of variance. The only exception is the USGS data set where only three pairs of samples are reported and thus the natural space numbers did not require transformation. Table 3 shows the results for the ICC analyses within laboratories, and also for the groups (Labs A, B, C, D) and (Labs A, B, C, D, H) aggregated. The variance components and p-values for the S-W normality test are also given. The lower part of Table 3 gives the aggregated results for the background samples only; Laboratories A and C did not contribute to these statistics but it is expected that they would perform similarly.

Table 3. Summary statistics for intra-class correlation coefficients.

All available Pairs

IntraClass Correlation Calculations: from SAS proc mixed
Log Space data

Lab	n obs	Int	Res	ICC	S-W p-value
A	6	7.6570E-01	5.4910E-01	0.5824	0.2786
A^	6	1.3310E+09	2.1025E+08	0.8636	0.7679
B	32	2.7493E+00	1.7470E-01	0.9403	0.1059
C	10	1.9870E+00	7.3050E-01	0.7312	0.0342
D	31	1.3966E+00	2.7860E-01	0.8337	0.8847
E	32	5.5988E+00	1.7150E+00	0.7655	0.6012
F	32	2.8559E+00	1.3172E+00	0.6844	0.6057
G	32	1.0962E+00	6.7860E-01	0.6176	0.8571
H	32	2.0647E+00	3.2810E-01	0.8629	0.1571
A,B,C,and D	80	2.0491E+00	3.0250E-01	0.8714	0.3296
A,B,C,D and H	112	2.0820E+00	2.8740E-01	0.8787	0.1540
^ natural space					

All NYC Background Pairs

IntraClass Correlation Calculations: from SAS proc mixed
Log Space data

Lab *	n obs	Int	Res	ICC	S-W p-value
A,B,C,and D	36	6.4570E-01	3.5070E-01	0.6480	0.2657
A,B,C,D and H	54	6.9800E-01	3.4290E-01	0.6706	0.2571

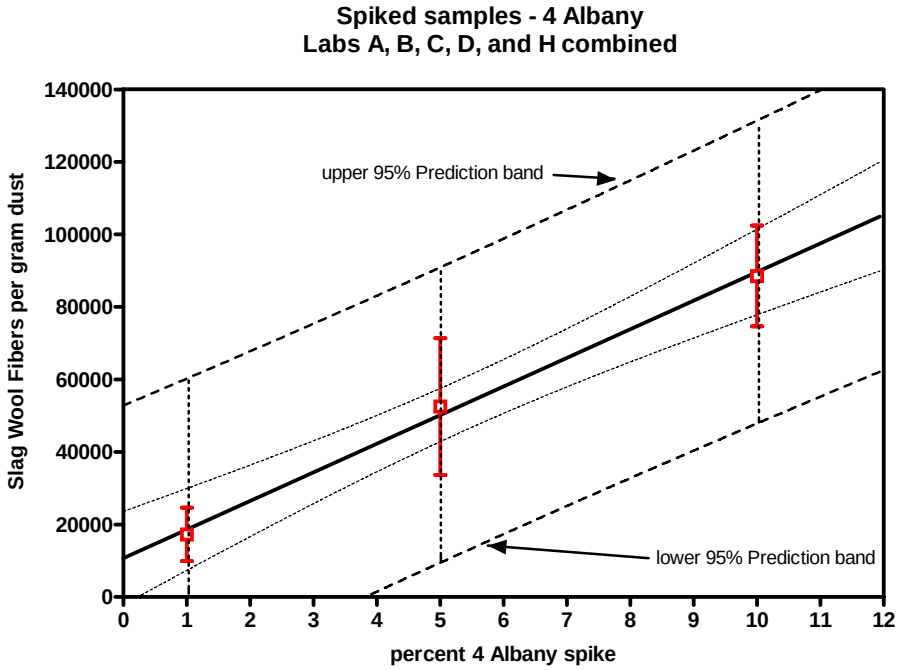
*Laboratories A and C did not report paired New York City background data

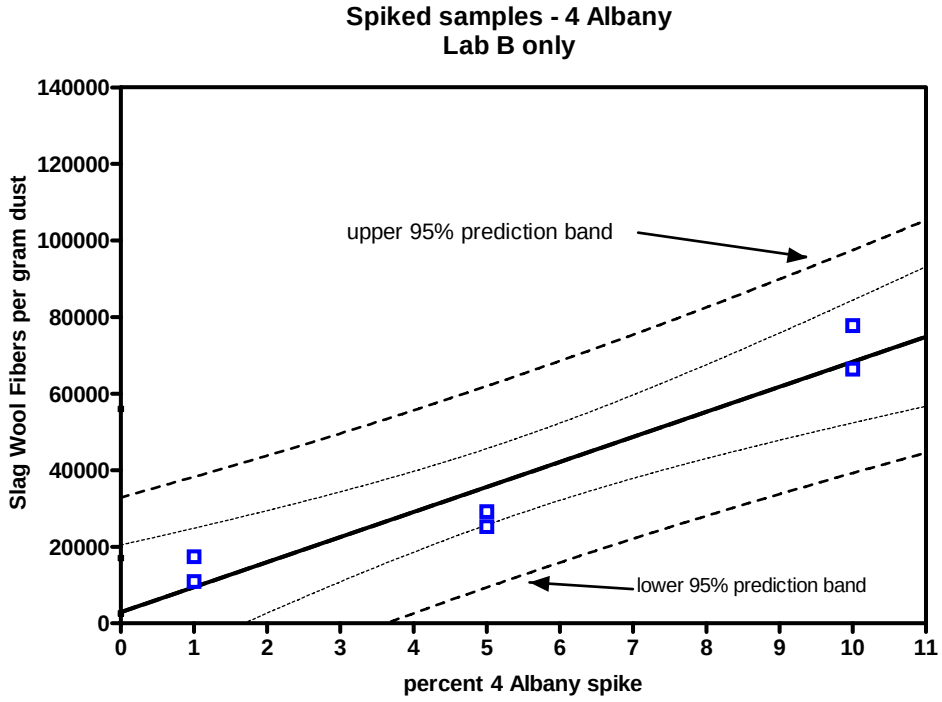
From this exercise, we see that all of the individual laboratories demonstrate reasonable ICCs (generally above 0.6). Furthermore, the laboratory groups chosen to demonstrate good validity show ICCs greater than 0.87 when all data are considered. When only the New York City background samples are analyzed, the ICCs are somewhat lower. These results can be interpreted to mean that about 35% of the variance is attributable to variability in the pooled laboratory analyses, and the remainder to true differences among the background samples.

As a further assessment of inter-laboratory precision, the between laboratory ANOVA shows no reason to reject the null hypothesis (Ho = no difference, in natural log space) among Laboratories A, B, D, and H. Laboratory C was left out of this analysis because they reported no background data at all.

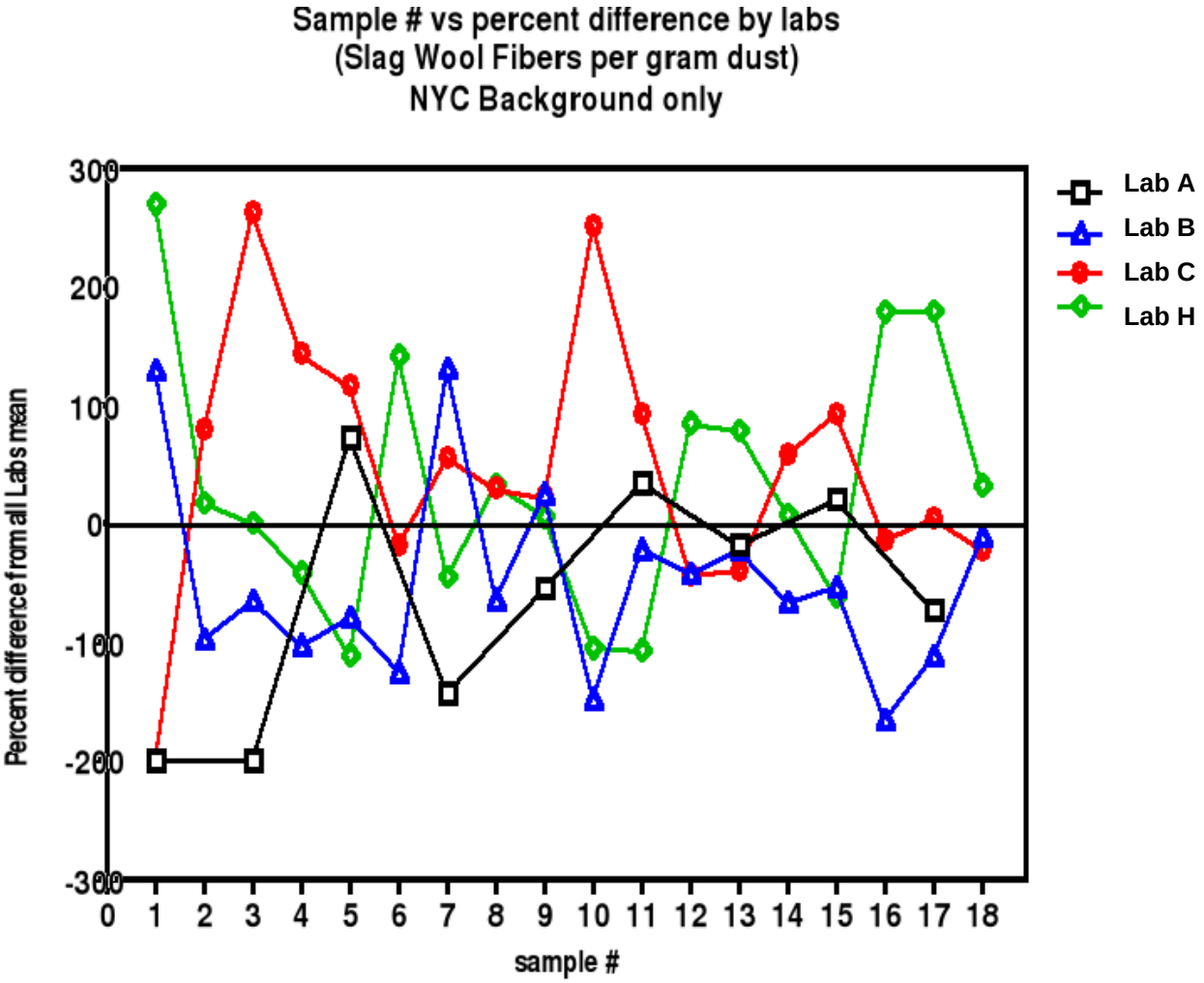
Evaluation of Unknown Samples

In the previous section we qualified a group of laboratories for measurement of unknowns based on spiked samples (validity), and comparative precision measures based on ICC and ANOVA. We now assume that these laboratories are statistically similar and combine their spike results into a single response graph. Based on these results, we calculate 95% confidence intervals and 95% prediction bands as illustrated in the figures below.





The major effort here is to estimate the performance of the aggregate laboratory group (A, B, C, D, and H) with respect to the group of samples from the greater New York City area designated as "background" or "non-WTC impacted". The composite behavior of these samples is illustrated below with respect to the analytical laboratories. The graph indicates no consistent (high or low) percent bias from the cross laboratory means. This confirms the conjecture made earlier that these laboratories are statistically similar. We caution that Laboratory C did not provide any background data at all and could not be directly included here, however, it is assumed that it would behave like the others.



The next step is to assess how an individual (presumably unknown) dust sample assay relates to the amount of spiked 4 Albany dust percentage. Given the graph and underlying statistics of the above figure entitled "Spiked Samples – 4 Albany, Labs A, B, C, D and H Combined), one can calculate the x-value in % spiked 4 Albany equivalent and the 95% confidence interval for the prediction for any unknown sample measurement from any laboratory. This is essentially the use of the prediction band graph above in reverse. As such the prediction of "x" and the CI take the following form:

$$X_{predicted} = (Y_{bar} - a)/(b)$$

$$CI = X_{predicted} \pm [t(RSE)/b] * \{1/m + 1/n + [(Y_{bar} - y_{bar})^2/(b^2(n-1)s_x^2)\}^{1/2}$$

where Y_{bar} is the mean laboratory measurement, a and b are the intercept and slope of the regression, t is the critical t-value for n-2 degrees of freedom, RSE is the residual standard error, m is the # of replicate measurements, n is the number of calibration points, y_{bar} is the mean of the regression y data, and s_x is the standard deviation of the x values of the regression data.

The reported slag wool results for the background samples can now be interpreted. Table 5 shows the results for each background sample measurement across all participating laboratories as a prediction of the percent equivalent 4 Albany spike level and half of the 95% confidence interval associated with the measurement. There are a total of 63 measurement results in the table.

Table 5: Results for each background sample across all participating laboratories as a prediction of the percent equivalent 4 Albany spike level and ± 95% confidence interval.

Lab*	A		B		D		H	
Sample #	Percent	CI +-	Percent	CI +-	Percent	CI +-	Percent	CI +-
1	-1.35	5.43	-0.89	5.40	-1.35	5.43	-0.69	5.38
2			-1.03	5.41	-0.47	5.37	-0.66	5.38
3	-1.35	5.43	-0.92	5.40	0.14	5.33	-0.71	5.39
4			-1.01	5.41	-0.14	5.35	-0.79	5.39
5	0.72	5.29	-0.43	5.37	1.05	5.28	-0.68	5.38
6			-0.38	5.36	1.00	5.28	3.06	5.20
7	-0.61	5.38	2.98	5.20	1.99	5.23	0.68	5.30
8			0.01	5.34	0.94	5.28	0.98	5.28
9	0.20	5.33	1.05	5.28	1.02	5.28	0.84	5.29
10			-0.88	5.40	2.68	5.21	-0.50	5.37
25	7.95	5.21	5.75	5.17	10.28	5.31	2.35	5.22
26			3.87	5.18	3.80	5.18	8.05	5.21
27	0.99	5.28	0.93	5.28	0.71	5.30	2.23	5.22
28			0.73	5.29	2.68	5.21	1.88	5.24
29	0.02	5.34	-0.44	5.37	0.47	5.31	-0.49	5.37
30			-0.88	5.40	1.21	5.27	3.86	5.18
31	3.88	5.18	2.29	5.22	7.07	5.18	14.25	5.63
32			4.79	5.17	4.41	5.17	6.18	5.17

*Laboratory C was left out of this analysis because they reported no background data.

We note that negative entries above are only statistical constructs. Of the 63 background measurements in this table, 7 (or about 11%) exceed the 4 Albany 5% spike level; 2 of the 63 measurements exceed the 10% 4 Albany spike level. If the upper confidence limits are considered, 42 out of 63 (67%) exceed the 5% spike level and 7 of 63 (11%)

exceed the 10% spike level. For instance for sample 25 at lab A the percent equivalent of the fiber measurement is 7.95% and the upper confidence limit is 7.95% + 5.21% = 13.16%.

As a further exercise, we calculated the same statistics for data within only one laboratory (choosing Laboratory B as the example); these results do not include scatter in the regression from the other qualified laboratories. Here we find some improvement: we see 3 of 18 values (16.7%) exceed the 5% 4 Albany dust level and 0 of 18 values exceed the 10% 4 Albany dust level. For the upper confidence levels, 6 of 18 values exceed the 5% 4 Albany dust level and 1 of 18 values exceed the 10% 4 Albany dust level.

Conclusions

The conclusions are based solely on the analytical data provided from the laboratory test and a few analyses of the 100% WTC spike samples. From validity estimates based on expected slopes and data scatter of WTC spiked samples, five of eight laboratories (A, B, C, D, and H) were used for further analysis. Intra-class correlation coefficients (with natural log transformation) for individual labs and for the group of five demonstrate similar and reasonable values (>0.7) when all available data are considered. One-way ANOVA analysis of Laboratories A, B, D, and H results provides no evidence to reject the null hypothesis (that the results are from the same distribution). Laboratory C was not included here because of insufficient reported data but, based on spike sample statistics, it is likely that that they too would fall into this category.

Under the practical constraints that the five laboratories are used at random with one analysis per unknown sample, we cannot expect statistical discrimination at the 1% or 5% 4 Albany spike equivalent level because the upper 95% prediction bounds exceeds the 5% spike equivalent level across the board. Reasonable discrimination is possible at the 10% 4 Albany spike equivalent level because the lower bound on10% equivalent measurements is approximately equal to the mean at 5% Albany spike equivalent level.